Bulletin 199

DEPARTMENT OF THE INTERIOR
ALBERT B. FALL, Secretary

BUREAU OF MINES
H. FOSTER BAIN, Director

# EXPERIMENTAL PRODUCTION OF ALLOY STEELS

BY

H. W. GILLETT and E. L. MACK

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# EXPERIMENTAL PRODUCTION OF CERTAIN ALLOY STEELS.

By H. W. Gillett and E. L. Mack.

## INTRODUCTION.

The production of small heats of alloy steels on an experimental scale is often desirable in beginning the study of new alloy steels before large amounts of expensive alloys are used in heats of commercial size. Such small heats can sometimes be made up at crucible-steel plants, but few crucible-steel makers care to undertake experimental heats for other firms. Small electric furnaces offer some advantages over crucible furnaces for experimental work.

Hansen [1] in 1909 described a small 100-kilowatt single-phase, direct-arc furnace with a capacity of 50 to 300 pounds of steel, and such furnaces, of 200 pounds capacity or larger, are being successfully used by various firms for experimental work. They have the advantage of using the same metallurgical procedure as is used in practice for larger electric furnaces. The very small sizes have the drawback of lack of precision in controlling the carbon content of the steel. Small Rennerfelt two-phase, indirect-arc furnaces of 40 to 60 kilowatts and small Industrial Electric Furnace Co. furnaces, single-phase with one electrode embedded in the hearth, of 25 to 50 kilowatts, are also in use by a few firms for experimental work or for production on a very small scale. Hoskins carbon-plate resistor crucible furnaces are also sometimes used. Little seems to have been published on the use of these furnaces in making experimental alloy steels.

Keeney [2] has given data on the making of uranium steels in a tilting Siemens furnace of 25 pounds capacity, and has described [3] a small stationary Siemens furnace.

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[1] Hansen, C. A., Small experimental Heroult furnace: Electrochem. and Met. Ind., vol. 7, 1909, p. 206.

[2] Keeney, R. M., Manufacture of ferro-alloys: Bull. Am. Inst. Min. Eng. Aug., 1918, p. 1321.

[3] Lyon, D. A., and Keeney, R. M., Melting copper ores in the electric furnace: Bull. 81, Bureau of Mines, 1915, p. 17.

More recently, two direct-arc furnaces for experimental use in the laboratory have been described,[4] and the Ajax Electrothermic Corporation[5] has brought out its high-frequency induction, eddy-current furnace, which has been developed mainly in small sizes, such as one to handle 25-pound charges of steel. This type should be well fitted for work on a very small scale and should allow satisfactory control of carbon, but is expensive.

The Bureau of Mines has recently made up experimental heats of alloy steels for the Army and the Navy.

The steels for the Army were desired for work on gun erosion, especially as regards the effect of nitrogen on the steel.[6] No physical tests of any of these steels were made by the Army.

The ingots made for the Navy were in two series, the first being rolled, heat-treated, and given physical tests by the Bureau of Standards. A report of the physical tests on these steels under definite heat treatment is given in Technologic Paper 207 of that bureau. Tests on steels of a similar type are also given by Johnson.[6a] The second series was made in larger ingots, which were rolled into plates, cut up into smaller plates, and heat-treated to different Brinell hardness numbers by the Halcomb Steel Co. Physical tests of this series under several heat treatments are being made by the Navy but are not yet finished.

Another series of alloy steels was made up in the course of cooperative work with the Vanadium Corporation of America and the Welsbach Co.; various impact and endurance tests are being made on these steels, the results to be given in a later publication. The present report deals only with the preparation of the ingots on any of the series of steels.

Some of the points brought out in preparing the steels, particularly as to the recovery of the alloying elements from the various ferro-alloys entering the steel, may, however, be of interest and are therefore put on record. The indirect-arc furnace finally used seems also to be a sufficiently useful piece of experimental apparatus to justify its description. There is, of course, nothing new about a furnace of this type, but it seems to fit in between the 25-pound high-frequency furnace and the 200-pound direct-arc furnace, being, in a 100-pound size, probably as satisfactory as either of the other types, and it can be constructed more cheaply than either of the other two. For a brief review of the ferro-alloy field, see Richards.[7]

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[4] Advisory Council of Science and Industry, Australia, Manufacture and uses of ferro-alloys and alloy steels: Bull. 9, 1918, pp. 36, 39.

[5] Northrup, E. F., High-frequency induction steel furnace: Chem. and Met. Eng., vol. 24, 1921, p. 309.

[6] Wheeler, H. E., Nitrogen in steel and the erosion of guns: Min. and Metal., April, 1920, No. 160, sec. 4.

[6a] Johnson, C. M., Some alloy steels of high elastic limit, their heat treatment and microstructure: Trans. Am. Soc. for Steel Treating, vol. 2, 1922, p. 501.

[7] Richards, J. W., Chem. and Met. Eng., vol. 19, 1918, p. 501.

### SYMBOLS FOR FERRO-ALLOYS.

Inasmuch as repeated reference will be made to various ferro-alloys, they will hereafter frequently be abbreviated to the following chemical symbols:

FeMn = Ferromanganese.
FeSi = Ferrosilicon.
FeW = Ferrotungsten.
FeMo = Ferromolybdenum.
FeU = Ferrouranium.
FeZr = Ferrozirconium.
FeTi = Ferrotitanium.
FeB = Ferroboron.
SiZr = Silicozirconium.
SiZr = Silicozirconium.

### ACKNOWLEDGMENTS.

Acknowledgment is due to Cornell University for the use, by a cooperative agreement, of its laboratories, which were especially suitable for these tests; to Prof. T. R. Briggs for his interest and cooperation; to A. E. Rhoads and C. E. Williams, of the Bureau of Mines, for aid in part of the work; to Lieut. L. S. Deitz, jr., and Lieut. J. G. Thompson, Ordnance Department, and to Mr. E. O. Denzler, Chemical Warfare Service, U. S. Army, who were assigned to duty at the Bureau of Mines during most of the work on the steels made at the request of the Army; to Lieut. S. W. Woodworth, U. S. N., who actively cooperated with the bureau, and to Lieut. J. O. Jenkins, U. S. N., and Lieut. R. McLane, U. S. N., Mr. W. B. Smith, C. G. M. U. S. N., and Mr. F. G. Dawson, C. M. M., U. S. N. R. F., who were assigned to duty at the bureau during part of the work on the steels made at the request of the Navy; to Mr. R. J. Thompson, metallurgist on the cooperative work with the Vanadium Corp., and to Mr. V. H. Schnee, chemist on the cooperative work with the Welsbach Co.; and especially to Dr. C. L. Parsons and to Dr. R. B. Moore, successively chief chemists of the Bureau of Mines, under whose direction all the work was done.

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It should also be stated that part of the supplies, such as ferro-alloys required in the preparation of steels for the Navy were supplied by the Navy. Aside from this and from the services of men assigned to the work by the Army and the Navy, the expenses of the work done for these departments were paid from the Bureau of Mines appropriation.

### REASONS FOR UNDERTAKING THE WORK.

In 1917 the Bureau of Mines received information from a creditable source that Germany was using uranium steel in the liners of some high-power naval guns. It was stated that uranium stiffens steel at high temperatures, and raises the softening point some 200° C., so that gun erosion is reduced. The fact that the German guns retained accuracy of fire at the end of the Jutland naval engagement was ascribed to the uranium-steel gun liners. The report agreed with previous less circumstantial reports. Somewhat similar reports had been received as to the use of molybdenum steel. Whether these reports had any foundation in fact is still unknown to the writers, but under the conditions existing in 1917 they seemed of sufficient importance to warrant an investigation.

The Bureau of Mines had some ferrouranium on hand from its work[8] on the production of that alloy, and because the Bureau of Mines had the use, at its Ithaca field office, of the electric-furnace equipment of Cornell University, which was suitable for the production of small experimental heats of steel, the bureau was requested by Col. T. C. Dickson, commanding the Watertown Arsenal, to prepare a series of uranium steels.

The series of uranium steels desired was to analyze as follows:

*Desired composition of uranium steel.*

| | Per cent. |
|---|---|
| C | 0.40 to 0.50 |
| Mn | 0.35 to 0.80 |
| Si | 0.15 to 0.35 |
| S | Not over 0.05 |
| P | Not over 0.05 |
| U | 0.5, 1, 1.5 per cent, etc., as far up as was feasible, in 0.50 per cent steps |

The steel was to be supplied in ingots, from which a sound bar 1 inch in diameter and 15 pounds in weight could be forged, this being enough for physical test specimens and for a pressure plug for an erosion test by the method of Langenberg and Fay [9]—inserting the plug in the mushroom head of a large caliber gun. Forging as well as testing was to be done by the arsenal.

The arsenal later requested that similar series of tungsten and molybdenum steels be supplied, the other elements being the same as in the uranium series, one series to contain 1 to 15 per cent W by 1 per cent steps, the other to contain 1 to 15 per cent Mo by 1 per cent steps. After the delivery of the steels was begun, the arsenal found it impossible to forge the ingots, so the bulk of the steels sent to the arsenal were forged at Ithaca into about 1¼-inch diameter bars before shipment.

When the work was begun the arsenal regarded uranium steel as the most important and asked that the uranium series be prepared first. After some work on this series had been done the arsenal came to the conclusion that it was doubtful if uranium steel really had been used by Germany and that the other series were more important. Work on the uranium series was therefore dropped, the tungsten series made, and the molybdenum series begun.

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[8] Gillett, H. W., and Mack, E. L., Preparation of ferrouranium: Tech. Paper 177. Bureau of Mines, 1917, 46 pp.; Ferrouranium, Jour. Ind. Eng. Chem., vol. 9, 1917, p. 342.

[9] Fay, H., Erosion and hardening of large guns: Iron Age, vol. 98, 1916, p. 1290.

The arsenal then requested that work on the molybdenum series be stopped and another set of steels be made up, as the plan of attack upon the erosion problem had been changed, and these were desired in the study of another method of testing than the pressure-plug method. These steels were to contain chromium, vanadium, titanium, nickel, and aluminum as alloying elements; some of the steels were to have but one, others up to four, of these elements.

All except two or three of these steels were shipped to the arsenal by November 8, 1918, but the armistice necessitated changes in the arsenal's plans for experimental work and no physical tests of the steels were made.

The Bureau of Mines has analyzed the Army steels for its own guidance only, for analysis of the steels for the arsenal was also scheduled to be made by the arsenal, though it was not, as it happened. Many analyses of the Navy steels for Zr, Ti, and Al were made by the Bureau of Standards. The analytical figures given herein on the first series of Navy steels are averaged from all the analyses by both bureaus. Analyses for Cr, Mo, and V in the third series of steels, except those also containing cerium, were made by the Vanadium Corporation.

## STEELS MADE BY MICHIGAN STEEL CASTINGS CO.

After the first laboratory steel furnace was built, many difficulties developed in connection with its use, hence advantage was taken of the offer of the Michigan Steel Castings Co., of Detroit, to make up a few steels. This firm was interested in uranium steel and had made a trial heat previously, analyzing 0.20 C, 0.65 Mn, 0.31 U, which showed up well.

The Michigan Steel Castings Co. made the steels by pouring about 100 pounds (estimated) of steel from one of the Heroult furnaces into a heated hand ladle and feeding the crushed ferro into the stream of metal. On account of the chilling effect of large amounts of ferro no attempt was made to prepare steels very high in alloying element.

The steel was poured into a baked sand mold, making an ingot 3¾ inches in diameter by 15 inches high. The balance of the metal was poured into "Keelblock" test-bar molds in sand. Drillings for analysis were taken from the riser of the test bar. The liquid steel used ran from 0.04 to 0.05 per cent in S and P. A small pellet of aluminum was used in each ladle for deoxidation.

TABLE 1.—*Results on making uranium steels.*

| M. S. C. test No. | Composition of liquid steel used. | | | No. of ferro.[a] | Composition of ferro used. | | | Ferro used. | Calculated composition of steel. | | | | Composition of steel by analysis. | | | | Apparent recovery of U.[b] | Remarks. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | C. | Si. | Mn. | | U. | C. | Si. | | C. | Si. | Mn. | U. | C. | Si. | Mn. | U.[b] | | |
| | *P. ct.* | *P. ct.* | *P. ct.* | | *P. ct.* | *P. ct.* | *P. ct.* | *Lbs.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | | |
| 11 | 0.28 | 0.28 | 0.58 | 34 | 33 | 4.8 | 0.8 | 1 | 0.33 | 0.29 | 0.57 | 0.33 | 0.33 | 0.26 | 0.48 | 0.30 | 91 | Ferro was apparently completely dissolved. |
| 12 | .28 | .28 | .58 | 45 | 39 | 5.2 | 1.6 | 1.67 | .36 | .31 | .56 | .66 | .32 | .30 | .39 | .45 | 68 | Some of the ferro seemed to be oxidized, as a scum formed on the metal. |
| 13 | .28 | .28 | .58 | 84<br>50 | 47.5<br>65 | 2.45<br>5.3 | 1.4<br>.5 | 1<br>.96 | .36 | .33 | .55 | 1.1 | .35 | .27 | .39 | .56 | 51 | Do. |
| 14 | .28 | .28 | .58 | 50<br>66<br>81 | 65<br>58<br>53.5 | 5.3<br>2.4<br>2.4 | .5<br>1<br>1.2 | .76<br>1<br>1.17 | .38 | .31 | .54 | 1.7 | .35 | .35 | .48 | .93 | 55 | Observers were not sure whether the ferro was completely dissolved or not. |
| 15 | .24 | .20 | .54 | 94<br>86 | 68.5<br>72 | 5<br>2.4 | .3<br>.6 | 2.15<br>1.62 | .37 | .22 | .52 | 2.63 | .33 | .37 | .48 | 1.83 | 70 | Ferro not completely dissolved. |
| 16 | .26 | .21 | .51 | 67<br>39 | 44.5<br>47.5 | 2.25<br>4.2 | 1<br>.3 | 6<br>[c]4.33 | .56 | .27 | .46 | 4.72 | [c].70 | .41 | .27 | 3.49 | 74 | (*c*). |

[a] See Gillett, H. W., and Mack, E. L., Preparation of ferrouranium, Tech. Paper 177, Bureau of Mines, for details of the making of these ferros.

[b] These figures are not necessarily correct, as there was undoubtedly segregation, the U analysis not necessarily being representative of the average U content of the ingot.

[c] 90 pounds metal poured into hot crucible instead of ladle, onto 10½ pounds ferro in bottom of crucible. Metal froze. Heated 4½ hours in oil furnace. Metal still rather cold. Carbon probably taken up from crucible.

### URANIUM STEELS.

The results on making uranium steels are shown in Table 1.

The Michigan Steel Castings Co. sent the test bars to the Standard Alloy Co. for determination of uranium and for physical tests. The uranium analysis by the Bureau of Mines on the sample taken from the riser and that by the Standard Alloy Co. on a sample taken from the test bar did not agree.

*Analyses of steel samples.*

| No. of sample. | Composition, per cent. | | | | | Uranium, per cent. | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | Standard Alloy Co. analyses of test bar. | |
| | C. | Si. | Mn. | P. | S. | Bureau of Mines analyses of riser. | First report, original method. | Second report, revised method. |
| 11 | 0.33 | 0.25 | 0.34 | 0.035 | 0.011 | 0.31<br>.30<br>Av. 0.30 | 0.26 | 0.49 |
| 12 | .32 | .30 | .38 | .036 | .015 | .41<br>.49<br>Av. .45 | .29 | .78 |
| 13 | .35 | .27 | .35 | .032 | .013 | .56<br>.57<br>Av. .56 | .39 | .45 |
| 14 | .35 | .30 | .38 | .036 | .016 | .97<br>.89<br>Av. .93 | .37 | .87 |
| 15 | .33 | .37 | .48 | .033 | .034 | 1.81<br>1.83<br>Av. 1.83 | ........... | 1.40 |
| 16 | .70 | .41 | .35 | ........... | ........... | 3.47<br>3.51<br>Av. 3.49 | ........... | n. d. |

The methods of analysis were then compared by interchanging samples of test bar 15.

*Analyses of sample 15.*

| Sample. | Bureau of Mines analysis. | Standard Alloy Co. analysis. | |
|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* |
| Bureau of Mines, original from riser | 1.83 | [a] 1.88 | [b] 1.69 |
| Standard Alloy Co., from one end of bar | .......... | 1.40 | .......... |
| Standard Alloy Co., from other end of bar | 1.06 | [a] 1.04 | [b] 1.02 |

[a] Using Standard Alloy Co. revised method, precipitation as phosphate, which is reduced and titrated.

[b] Using Bureau of Mines method, precipitation as oxide, gravimetric method.

This comparison of results, compiled after a number of uranium steels had been made up and shipped, shows that there was material segregation of uranium in the uranium steels made by the Michigan Steel Castings Co.

The physical properties of the test bars were reported by the Standard Alloys Co. as follows: All samples quenched at 1,620° F. (883° C.), samples M drawn at 800° F. (427° C.), samples N drawn at 1,200° F. (647° C.). Standard 0.5-inch diameter, 2-inch length test bar used.

*Physical properties of test bars.*

| No. of sample. | Tensile strength. | Yield point. | Elongation. | Reduction of area. | Fracture. |
|---|---|---|---|---|---|
| | *Lbs. per sq. in.* | *Lbs. per sq. in.* | *Per cent.* | *Per cent.* | |
| 11 M | 121,800 | 89,750 | 9 | 34.4 | Fine, silky half cup. |
| 11 N | 85,600 | 65,600 | 20.5 | 54.6 | Fine, silky cup. |
| 12 M | 149,800 | 141,700 | 1.5 | 4 | Coarse, granular, ragged. |
| 12 N | 93,800 | 79,300 | 13 | 21 | Do. |
| 13 M | 148,000 | 100,800 | 8.5 | 29.2 | Fine, silky half cup. |
| 13 N | 95,600 | 81,500 | 17.5 | 40.2 | Do. |
| 14 M | 126,800 | 79,900 | 1.5 | 4 | Coarse, granular, ragged. |
| 14 N | 90,000 | 76,000 | 10 | 14.2 | Do. |
| 15 M | 96,400 | None. | None. | None. | Coarse, granular, straight. |
| 15 N | 84,900 | None. | 1.5 | 1.2 | Coarse, granular, ragged. |

According to the Standard Alloys Co., the greatest benefit claimed for uranium in low-carbon steels is from the addition of not over 0.3 per cent U.

Several tungsten and molybdenum steels were made up at the Michigan Steel Castings Co. The ferro-alloy was preheated in a crucible furnace, molten steel then added, and the mixture heated in order to produce a molten rich W or Mo alloy that could be added to ladles of carbon steel taken from the Heroult furnaces; however the maximum temperature attainable in the oil-fired crucible furnace was too low to produce a sufficiently fluid rich alloy and this method had to be abandoned. A 1 per cent Mo steel was satisfactorily made up by adding the ferromolybdenum in the ladle, but the steels of higher Mo and W content desired by the arsenal could not be so made on account of the chilling of the steel when large amounts of solid ferro are added to the ladle.

The Michigan Steel Castings Co. made every effort to prepare these steels, but the futility of attempting to make laboratory-sized heats with equipment designed for large-scale production soon became apparent, and the additional work was done in the laboratory, starting with a small direct-arc furnace.

## USE OF SMALL DIRECT-ARC FURNACE.

Since the arsenal required but 15 pounds of sound forged bar of each composition, it was thought that a 30-pound charge should allow for melting losses and for cropping the ingot free from pipe. A single-phase, direct-arc type of furnace was chosen. A rectangular shell 11½ by 15 inches by 13 inches high was mounted on trunnions and counterbalanced to tilt about the spout for direct pouring, since it was feared that with so small a charge it would not be practicable on account of too rapid chilling of the metal to pour first into a ladle and then into the mold.

Inasmuch as the water-cooled hearth of the furnace used for making ferrouranium had proved satisfactory, it was decided to test a thin refractory lining and to try to maintain this by water-cooling; therefore the furnace was lined with split magnesite brick 1¼ inches thick, save for the upper course, which was 2½ inches thick, making a chamber 12½ inches long by 9 inches wide and 13 inches high. The bottom was built up into hearth form by ramming magnesite, bonded with water glass, into the corners.

Two perforated spray pipes were placed about the furnace, one at the top and one about halfway up, so that the sides and bottom were covered with a uniform film of flowing water which dripped off the bottom into an open tank placed beneath the furnace, from which it was led to the sewer. A shell was welded about the spout to prevent water from getting into the mold when the furnace was tilted to pour. A pouring spout measuring 2 by 4 inches after it had been lined with split brick was provided. The roof was made of split zirkite brick held in an iron frame set on top of the furnace body. Holes were ground out in the zirkite brick for passage of the two 2-inch diameter graphite electrodes, which were on the center line of the furnace about 2 inches apart. The electrodes were thus 3½ inches from the side walls. The water coolers through which the electrodes passed with one-sixteenth-inch clearance were large enough to cover nearly all of the roof and to cool it as well as the electrodes.

The electrodes were supported by masts attached to the furnace and were adjusted by handwheels. Connection was made to the flexible leads by electrode holders, which were uncooled as the roof coolers were sufficient.

The furnace took a current of 500 to 700 amperes at 75 to 90 volts. The average load was about 40 kilowatts at a power factor of about 0.90.

The split cast-iron ingot mold gave an ingot 2½ by 2½ inches at the butt, 3 by 3 inches at the top, and about 16 inches tall. As a 30-pound charge scarcely filled this and the furnace would not hold much over 30 pounds, a hot-top brick could not be used.

When the metal was poured direct from furnace to mold it was poured through a conical alundum-lined funnel with a one-half inch hole in the bottom, set on the mold, to direct the stream.

Experiments were tried with making up the series of tungsten steels in 30-pound heats, charging iron, ferrotungsten, and washed metal with part of the ferrosilicon and ferromanganese at the start, plus about 1¼ pounds of a mixture of 75 per cent CaO, 20 per cent $SiO_2$, 5 per cent $CaF_2$ for slag, and adding the balance of the ferrosilicon and ferromanganese, after the charge was fully melted, and later, just before pouring. After the slag was skimmed off, the metal was poured, at first without allowing time for the metal to cool and give up dissolved gases, which produced unsound ingots at any of the high pouring temperatures tried, even though as much as 0.05 per cent aluminum was added just before pouring. The metal was, in later runs, poured into a preheated ladle, thence to the mold. By using lower pouring temperatures sound ingots were obtained.

### CONTROL OF CARBON CONTENT.

The main trouble, however, was in controlling the carbon content of the steel. One charge calculated for 0.35 per cent C gave 0.7 per cent C, two each calculated for 0.7 per cent C gave 0.36 per cent and 0.97 per cent C, and one calculated for 0.61 per cent C gave 0.32 per cent C.

On account of the necessary clearance between electrodes and the electrode coolers in the roof, the furnace could not be tightly closed against the entrance of air. For this reason, as well as for the presence of a little scale on the iron used, some decrease in the carbon found from that calculated was to be expected. In the great majority of melts, however, there was a decided pick-up of carbon, which came, of course, from the electrodes. It soon became evident that the carbon pick-up was related to the way the arcs held during the melting-down period, when the charge was still solid, and before a sufficient pool of metal and slag had formed beneath the electrode to sustain the arc. If the charge happened to be piled so that, as it melted down, the arc broke often and was " snappy," contact had to be made often with the charge to restart the arc. During each contact carbon was absorbed from the electrode. If, on the other hand, the charge happened to lie so that the arc was smoother and broke less often, less carbon was absorbed. In a furnace of commercial size, variations in the amount of carbon so absorbed would be negligible in comparison with the whole charge, but on a 30-pound charge they were not. The electrode loss varied from one-half to 1½ inches per electrode per heat—that is, 0.2 to 0.6 pound graphite. Attempts were made to control the carbon content by sampling, after the charge was melted, making a rapid carbon analysis, and readjusting the composition of the charge, but the time was so short and the difficulty of readjusting the charge so great that this method appeared impractical.

Such a tiny direct-arc furnace might be useful for university instruction, to show the way that type works, but it seemed obvious that to be of service in preparing steels of definite analysis the size of the furnace would have to be increased. The use of this furnace was therefore finally abandoned. While the next furnace was being constructed, however, work was continued with the direct-arc furnace, a few molybdenum and uranium steels being made.

## REFRACTORIES.

Various refractories were tried in the side walls above the slag line. Magnesite brick spalled rather badly, as would be expected in a furnace used intermittently, with one face of a 1¼-inch split brick at steel-melting temperature and the other face against the water-cooled shell. Zirkite did better, lasting 14 heats, but melted away a little and went into the slag. A purer zirconia refractory than that of zirkite, which was made from the natural unpurified ore, might be expected to do better. Bauxite brick melted away rapidly. Carborundum brick stood up well, but spattered globules of steel shot up against them and the C and Si content of the steel was thereby slightly raised. If used where such spatter can not reach them, they should give good service.

Considering the nearness of the arcs to the side walls, it appeared that the water-cooling gave the thin zirkite lining a much better life than would have been expected from a thicker lining not water-cooled.

The magnesite hearth stood up well enough with occasional patching, while hot, with crushed magnesite moistened with dilute water-glass. The zirkite roof bricks stood up satisfactorily, showing little tendency to soften or to crack. .

The recovery of W and Mo was found to be quantitative, all that was added being recovered in the steel. The recovery of uranium was not quantitative.

*Recovery of uranium.*

| Analysis of ferrouranium. | | | Per cent U— | | |
|---|---|---|---|---|---|
| C. | Si. | U. | Added. | Found. | Apparently recovered. |
| *Per cent.* | *Per cent.* | *Per cent.* | | | |
| 5 | 0.6 | 49.5 | 1.1 | 0.7 | 63 |
| 5 | .6 | 49.5 | 1.1 | .7 | 63 |
| 5.8 | .5 | 52 | 1.55 | .72 | 46 |
| 2.1 | 1.8 | 48 | 1.05 | .31 | 30 |
| 2.1 | 1.8 | 48 | 1.05 | .52 | 50 |

About one and a half minutes elapsed in these runs between the addition of ferrouranium, crushed to pea size, to the charge in the furnace, and pouring the melt into the mold direct from the furnace for the first three tests, or into the ladle for the last two tests. The slag was thoroughly skimmed off before the ferro was added and the ferro stirred in at once. The melt was held in the ladle about two minutes.

The samples for analysis were taken from the top of the ingots, and as previously stated but ascertained after these were shipped, segregation is to be expected, so that the recovery figures are not necessarily correct.

## USE OF INDIRECT-ARC FURNACE.

Since it was necessary to discard the small direct-arc furnace and either to build a larger one that would take a large charge and a longer time per heat, or to utilize a type in which there would be less contamination by the electrodes, it was decided to try an indirect-arc furnace, in which no slag need be used (thus tending toward better refractory life) and in which the charge could be melted down without contact with slag or electrodes. The metallurgy of the process would then be similar to that of crucible-steel melting, though done in an electric furnace.

For these reasons it was decided to try a few heats in the laboratory rocking brass-furnace.[10] A facing of magnesite cemented with water glass was put in after some of the regular brass furnace lining was chipped out and all brass shot were removed. A lining so put in could not be expected to wear well, but was hoped to show how the furnace would handle steel. Charges of 35 to 45 pounds were used instead of the 28 to 30 pound charges used in the direct-arc furnace.

The molds, which were washed over smoothly with thin alundum paste, dried, baked by playing a large gas flame on them and smoothed with fine sandpaper, were thereafter lightly smoked with a smoky kerosene flame.

### URANIUM STEELS IN RELINED ROCKING BRASS FURNACE.

In heat 1044 the furnace was preheated with 15 kilowatt hours, then all the 35-pound charge added except the ferrouranium, 50 grams of FeSi, and 5 grams of Al. After 27 kilowatt hours the metal was skimmed of the fallen pieces of magnesite lining, 400 grams of pea-size FeU (No. 77) containing 45½ per cent U, 2 per cent C, 4 per cent Si, and the FeSi, were added, 1 minute 10 seconds elapsing after adding the ferro and stirring it in before pouring into the ladle. Aluminum was added in the ladle. The metal was cold and skulled a good deal. The ingot was sound and forged well.

*Analysis of product of heat 1044.*

| Constituent. | Calculated. | Found. |
|---|---|---|
| C | 0.76 | 0.52 |
| Si | .45 | .18 |
| Mn | .94 | .62 |
| U | 1.15 | .44 |
| Al | .03 | n. d. |

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[10] Gillett, H. W., and Rhoads, A. E., Melting brass in a rocking electric furnace: Bull. 171, Bureau of Mines, 1918, p. 25.

Heat 1045 was run about the same, but as 600 grams of No. 77 FeU was to be used, the furnace was heated seven minutes after the FeU was added and before pouring. As the metal was too cold on the previous heat, 20 kilowatt hours were used on the preheat, 24 kilowatt hours before adding the FeU, 5 kilowatt hours after adding it, 49 in all. The ingot was satisfactory.

*Analysis of product of heat 1045.*

| Constituent. | Calculated. | Found. |
|---|---|---|
| C | 0.72 | 0.53 |
| Si | .44 | .29 |
| Mn | .94 | .63 |
| U | 1.69 | .04 |
| Al | .03 | n. d. |

In heat 1046 a 44-pound charge was used, 20 kilowatt hours preheat, 34½ kilowatt hours before adding FeU, when 665 grams No. 77 FeU were added together with a little FeSi and FeMn, 6 kilowatt hours more then used in about 10 minutes, and a further addition of 225 grams No. 28 FeU (35.5 per cent U, 4.9 per cent C, 0.5 per cent Si) made before pouring into the ladle. The ingot was sound.

*Analysis of product of heat 1046.*

| Constituent. | Calculated. | Found. |
|---|---|---|
| C | 0.68 | 0.60 |
| Si | .50 | .40 |
| Mn | .96 | .77 |
| U | 2.1 | .35 |
| Al | .03 | n. d. |

In these three heats the furnace was rocked by hand during the heat, starting with a small rocking arc as soon as the metal started to melt and steadily increasing the angle through which the furnace was rocked until when all the metal was melted, the whole lining, save the ends and the portion close to the charging door lining, was washed by the metal.

In the direct-arc furnace an apparent recovery of 30 to 60 per cent of the U added was made; in heat 1044, a 58 per cent recovery. In both tests the FeU was added just before pouring. In heat 1045 the ferro was allowed to remain in the furnace seven minutes before pouring, with an almost total loss of U. In heat 1046 FeU was added both before the heat was finished and at the very end. On the basis of the total U added, the apparent recovery was only 17 per cent, while if it be assumed that the U added before the heat was over was lost, as in heat 1045, the apparent recovery on the U added at the end is 70 per cent. The term "apparent recovery" is used because the liability to segregation of U in U steels was not established until these ingots had been forged and shipped.

The magnesite lining did not adhere well to the old lining, and no additional runs were made in this furnace shell. The loss of C, Si, and Mn, while not entirely uniform, showed a much better approach to it than in the direct-arc furnace and it was felt that for the purpose in hand the indirect-arc furnace was better fitted than the direct-arc type, hence an indirect-arc furnace was built. At this time it was not expected that any steels other than the U, Mo, and W series would be made, for the arsenal had not yet requested the others; therefore the furnace was built up as cheaply as possible, and designed for a charge of about 40 pounds.

## DESCRIPTION OF INDIRECT-ARC FURNACE.

A cylindrical sheet-iron shell 30 inches long by 30 inches diameter was bought at a junk shop. Two hoops of one-half by one-half inch stock were made, slipped over the shell while hot, and riveted thereto about 2 inches from each end, thus forming the rings supporting the shell. Each ring rested on two rollers held in a framework forming the base of the furnace. (See Pl. I, *A* and *B*.)

The shell was a horizontal cylinder, resting on four rollers beneath it. The braces on the base were so spaced and bent as to allow the shank of the pouring ladle to pass beneath the furnace. The furnace was raised enough on the supports to allow the metal to drain completely from the spout into a crucible used as a ladle.

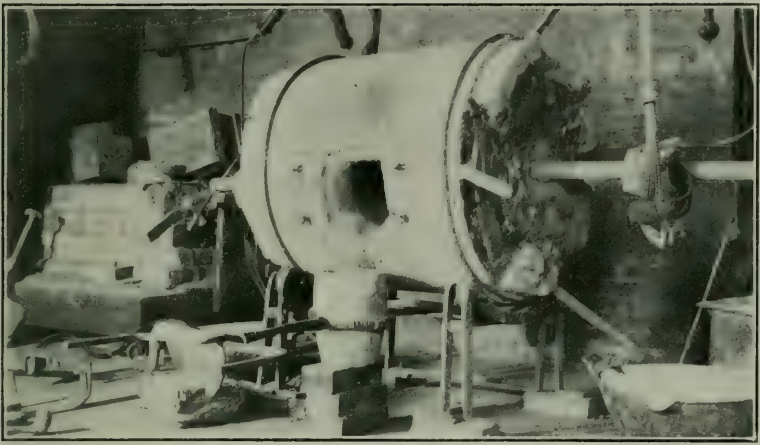
A hole 12 by 12 inches was cut in the shell for a combined charging door and pouring spout.

Four handles extending from the end of the shell at 45°, equally spaced, tilted the furnace.

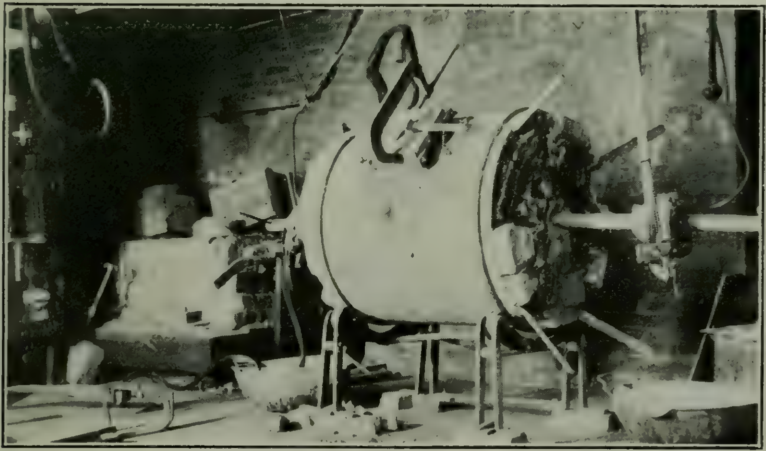
At one end, braces projecting from the end of the shell held an alberene slab 1 inch thick, through which a $2\frac{1}{16}$-inch hole was bored. This slab, 6 inches from the end of the furnace, supported a long electrode and took the bending strain. The 2-inch diameter round graphite electrode rested in this support as well as in the electrode hole in the end wall.

### ELECTRODES.

Two electrodes 40 to 48 inches long were used, one entering the furnace horizontally from each end, an electrode being started in service on the end with the alberene support. When it had worn off to a length of about 30 inches, it was transferred to the other end of the furnace, on which no other support than the refractory wall was provided, the shorter electrode being balanced well enough in and supported by the end wall alone. It could then be used until it had worn down to a length of about 20 inches and was then put in stock, as the short electrodes are useful in other work. The alberene



*A*. LABORATORY INDIRECT-ARC STEEL FURNACE, OPEN FOR CHARGING.



*B*. LABORATORY INDIRECT-ARC STEEL FURNACE, CLOSED FOR OPERATION.

support could be removed when it was desired to use short electrodes in both ends. These supports were finally abandoned in favor of a thick end wall, 15 to 18 inches, which supports even a very long electrode without any other support than the furnace. Jointed electrodes were used when the longer electrodes were not obtainable, but were far more likely to break; therefore, even had there been no use for the discarded butt, the long electrodes were no more expensive to use in the long run than jointed electrodes, with far less danger of spoiling a heat through breakage.

Flexible copper leads were bolted to split bronze electrode holders, fitting the electrodes tightly. The holders were not water-cooled, as they would have been more expensive to make, heavier, and more likely to cause breakage of the horizontal electrodes. Little or no cooling was needed on long electrodes, since the $C^2R$ heating was not great, as care was taken to make good contact between holder and electrode with copper gauze. When an electrode had worn down very short, the conducted heat made it desirable to cool the holder. In such cases, the holder was wrapped in asbestos cloth and water was dropped on it slowly from a hose whose end was suspended over the holder. The drip was caught in a large pan set on the floor and emptied when necessary. This cheap and crude method of cooling was entirely adequate for the purpose.

A hole was bored in the outer end of the electrode and an iron rod stuck through the hole to form a handle for the adjustment of the electrode. The length of the arc was thus controlled by hand, the electrode staying where it was put, as it fitted snugly into its hole. After the first few minutes' heating of a cold furnace, the arc requires little adjustment.

Since the electrodes became pointed by oxidation, due largely to the removal of the electrodes at the end of a heat to allow skimming before pouring, and since the alundum lining softened somewhat, it was necessary to punch out the electrode holes with a 2⅛-inch diameter steel bar immediately after pouring. This bar had a handle on one end like that used on the electrodes, and could readily be forced in and turned around to keep the electrode holes smooth and true. When this was done, very little patching of the electrode holes was needed.

The furnace lining consisted of 2½ inches of corundite fire brick next to the shell and at the ends, then of 4 inches of refractory brick, zirkite at the start, magnesite later, the latter being brought out clear to the shell about the combined charging door and pouring spout. Since the zirkite brick were made from crude zirkite ore and were plastic at the temperature attained, and since magnesite spalls badly when used intermittently at these temperatures, it was necessary to protect both with an inner layer of albundum cement.

### ROOF OF FURNACE.

A roof of magnesite bricks spalled so much as to require frequent patching. A zirkite brick lining made from natural zirconia ore softened much more than alundum and made too heavy a slag. A partial lining of refined zirconium oxide (about 99.5 per cent $ZrO_2$) bonded with a little water-glass was tried, and might perhaps have been satisfactory had it been used exclusively, but when in contact with alundum it fluxed down and was far less refractory than the impure zirkite. Aluminum oxide therefore appears to lower the melting point of zirconium oxide appreciably. The carborundum (Carbofrax) brick used as a door stood up so remarkably well, that the 4-inch magnesite backing to the inner alundum lining over the upper half of the furnace was replaced with 1¼ inches of magnesite, next the corundite and 2½ inches of carborundum (Refrax) brick over the magnesite when the second series of zirconium steels was made. The lower half of the furnace was left as before, the carborundum being used only in the upper part of the furnace where the lining never came in contact with the steel, since a crack in the alundum lining might have allowed the charge to come in contact with the carborundum. For fear that the Refrax brick might spall and allow carborundum to drop into the melt, a coating of alundum was maintained over the carborundum bricks, but this had to be patched often to maintain it at required efficiency.

When carborundum was used in the roof, all heats were made on the hearth proper. When magnesite or zirkite was used, less patching was required if the furnace was turned over between heats, making the roof for one heat the hearth for the next.

The Refrax (pure carborundum) brick were absolutely unharmed after some 40 heats when they were replaced by Carbofrax (clay-bonded carborundum) brick which had lower heat conductivity. These were put directly against the corundite backing, allowing the roof to be brought a little farther from the arc. After about 40 heats, the Carbofrax bricks appear unaffected, as far as can be seen without taking them out of the furnace, which is still in service. When the Carbofrax bricks were put in the roof and upper side walls, an attempt was made to coat them with a layer of pure electrically sintered, finely ground magnesite, bonded with a little dextrin. The magnesia lining, baked down to a solid mass of about the texture of an ordinary brick, showed only a slight tendency to crack. It did crack a little, yet that little was sufficient to make the lining come off, because it did not adhere to the Carbofrax brick back of it. It seems possible that a very thin layer of ordinary fire clay over the Carbofrax, followed by the magnesia layer, might fuse and stick the magnesia layer to the Carbofrax layer without reducing the refractoriness of the magnesia layer. The magnesia was sufficiently coherent, but failed to adhere to the Carbofrax.

The alundum coating was therefore used over the Carbofrax to prevent possible contamination of the charge by spalling of the brick or spatter of the steel against it.

From the behavior of the bare Carbofrax door, which is still in good condition after 200 heats, notwithstanding some rough treatment, the alundum layer over the roof may be a needless precaution. It was used because little more time was needed to patch the roof as well as the hearth when patching was required.

## SATISFACTORY FURNACE LINING.

The most satisfactory lining for such a furnace would appear to be as follows: Outside layer, Sil-o-cel or some similar reliable low-temperature insulator; second layer, high-grade fire brick, preferably one high in alumina, such as corundite; third layer, magnesite brick in the lower or hearth half, bare Carbofrax in the upper or roof half; and, as the inner layer in the hearth, electrically sintered, dextrin-bonded magnesia, if slags are to be taken off. If, as in the present tests, no refining is to be done, the hearth can be lined with alundum, which, although less refractory than the magnesia, softening a good deal at operating temperatures and requiring continual repair, is nevertheless not so likely to crack and let the steel work down into the hearth. If the furnace is to be used on steels of similar composition, so that metal retained in the hearth from one heat does not introduce contaminating elements into the next heat, a magnesia hearth would be preferable, but when, as in this work, successive heats of different composition have to be made, alundum appears, on the whole, to be better. This alundum lining, 1 to 2 inches thick, was rammed over the whole inner surface in layers when a fresh lining was put in, each layer being dried thoroughly with a gas flame, then moistened with a thin wash of alundum before a fresh layer is placed and dried. The corners were filled up with alundum and care taken to shape the hearth and pouring spout so that metal would drain completely. The furnace chamber proper then had an approximately spherical form of about 14 inches diameter.

When larger charges were required for later work the magnesite-brick backing to the hearth was made a little thinner, and by scraping out all slag immediately after pouring, and by filling up the hearth to its normal position only when patching, it was found possible to handle 100-pound charges without trouble if the charge was compact. "Armco" iron cut into 2 by 4 inch pieces from one-half-inch plate formed a sufficiently compact charge to be piled in the furnace without touching the electrodes.

At first the electrodes were mounted with their centers 2 inches below the center line of the furnace, bringing the 2-inch electrode 5 inches from the hearth and 7 inches from the roof to protect the roof somewhat and to bring the arc nearer the charge. This arrangement was satisfactory when very heavy scrap, such as crop ends of ingots, formed a large part of the charge, but made it hard to pile the charge in without touching the electrodes when light material only was used. This arrangement was retained during the manufacture of most of the steels which were cropped and forged at Ithaca for the arsenal and the crop ends utilized. When the steels for the Navy that were shipped without cropping were made, these larger heats being made up from new metal, it was necessary to locate the electrodes centrally to avoid piling the charge so near the electrodes that there was danger of pieces falling against them as the charge melted down, with a consequent pick-up of carbon due to this contact.

It was hoped that the furnace might be rocked when in operation, thus washing the refractories with the metal and thus increasing their life; however, the furnace was so heavy when lined up that without going to the expense of installing a motor-driven rocking mechanism, or at least providing ball-bearing rollers, it could not be rocked by hand without too great exertion, although it could be tilted by hand for pouring. Had it been expected that the furnace would be stationary when operated it would have been designed with a larger diameter to bring the arc farther away from the roof.

## REPAIRING FURNACE.

So small a furnace, with the refractories so close to the arc, used at extremely high temperatures, as it had to be, was known to require constant patching. The temperatures often used were decidedly higher than those of a large commercial steel furnace. First, it was necessary to get a small charge hot enough to pour from a ladle, which, though preheated, was nevertheless at a much lower temperature than the metal and chilled it a good deal. Second, when steels are made with such ferro-alloys as ferrouranium and ferrozirconium, where the recovery of alloying element is too low unless the ferro is added to superheated metal at the end of the heat, the steel must be very hot, especially when steels high in the alloying element are to be made up. Since there is grave danger of oxidation when these alloys are preheated, they were normally added cold and in attempting to make steels high in U or Zr under these conditions as much as 5 or 6 pounds of ferro was sometimes used in a total charge of 40 to 50 pounds.

When used in the production of plain carbon steels, or of such alloy steels as nickel, tungsten, chromium, or molybdenum, when the alloying element or the ferro-alloy can be added with the rest of the charge at the beginning of the heat without loss, the furnace, even as built, required only a very reasonable amount of patching to the alundum coating,[11] such as any laboratory furnace might be expected to require. Hansen states that he used alundum as a lining for a steel furnace (probably a direct-arc furnace, using a slag) with "disastrous results." It should therefore be emphasized that alundum is suitable only when simple melting, without refining, is to be done. Under the service conditions, extremely hot metal being required on perhaps half of the heats to take large alloy additions into solution, and the lining being chiseled free from adhering metal from the previous heat whenever a change of composition was made in the steels being produced, the refractory brick lining, whether zirkite or magnesite, had to be replaced about every 100 heats.

Inasmuch as continuous operation was not required, it was seldom that more than two heats were made without allowing the furnace to cool and making such repairs to the lining as were needed to keep the lining from serious injury. In the early part of the work it was especially necessary to know the analysis of each heat or each short series of heats in order to be able to calculate the next charges from the results of earlier ones as to losses of the various elements. More heats could be made with the furnace in a given time, even on intermittent operation, than the analytical staff could handle in the period.

Since intermittent operation only was contemplated, no heat-insulating brick such as infusorial earth brick were used. The hand could, nevertheless, be held on the shell at the end of a second heat; so although the furnace would not serve in the dimensions given for continuous operation it was fairly satisfactory, if continuous patching was done, for the conditions that had to be met.

## USES OF ALUNDUM CEMENT.

The charging door of the furnace was closed by a 9 by 10 inch plug-type door, made up of carborundum brick cemented together by and coated with alundum cement, and held in an iron frame and by a large clamp serving as a handle. The door was set in place and a bar thrust through two wire loops, one on each side of the door. The loops were held by eyebolts passing through the shell, and the door was tightened by putting an iron plate on the back of it to protect the bricks and then driving an iron wedge between this plate and the bar. The door was then luted in place with alundum cement in order that, after the furnace had become heated up sufficiently to hold the arc without breaking, a little thin alundum wash brushed over any cracks that developed in the luting and over the edges of the electrode holes (if any light from the arc showed between electrode and hole) served to keep the furnace tight, without any flame coming from any part of the furnace.

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[11] Hansen, C. A., Discussion: Trans. Am. Electrochem. Soc., vol. 17, 1910, p. 134.

The door only entered the charging opening for about half its thickness, its inner face being thus about a foot from the arc. There was some heat loss from a door made of so good a conductor of heat as carborundum and the door became red hot at the end of a heat, but its brightness served as a fairly accurate indication of the temperature of the furnace.

The door was removed and set aside while pouring, and was thus subject to violent changes in temperature as well as to being accidentally dropped. The durability of the doors was nevertheless remarkable, one giving over 100 heats and then failing only because it had been dropped too often, and another being still good after about 275 heats. The efficiency of carborundum for this purpose, as well as its showing in the direct-arc furnace, make it seem well worth a trial in the roof of a commercial steel furnace.

Alundum cement was indispensable as a material for the inner lining. Various magnesite-alundum and zirkite-alundum mixtures were tried for lining and patching, but nothing adhered so well as the plain alundum, nor did additions appear to improve the refractoriness. If care was taken to paint the places to be patched with a thin wash before ramming in thick cement after steel shot and badly slagged lining were cleaned out, quite large patches could be successfully made; especially could this be done if the furnace was so warm from the previous run that a person's hand could barely be kept inside it, and provided that, in preheating the furnace to operating temperature before charging, any large patch was baked in, with the furnace so placed that the patched spot was on the bottom.

The cylindrical form of the furnace makes this easy, for if there is a bad place in the roof to be patched that spot can be put at the bottom and the patch held in place by gravity until it is thoroughly baked, whereas if the same place had to be patched in the roof of a furnace not capable of being rolled completely over, the patch could not be made to stick.

Alundum cement was used for painting the molds before they were smoked. In early runs of the direct-arc furnace some of the molds were quite deeply eroded by the stream of metal striking the side. By filling up these rough spots with alundum cement and baking it in, the molds were kept in service. The crucible used as a pouring ladle was lined with a quarter-inch layer of alundum cement to prevent the steel from taking up carbon from the ladle, to allow a better retention of heat by the ladle than would have been possible with the bare crucible, and to form a layer that could be chipped out to free a skull that could not have been removed without breaking the crucible if there had not been such a lining. The alundum-lined ladle was preheated by directing inside of it a large gas flame or one from a Hauck kerosene burner. This preheat usually was started simultaneously with the steel furnace. At the time of pouring, the ladle was ordinarily at about 800° C. An iron ladle lined with alundum would perhaps have been better than the crucible.

Alundum cement was used for luting the door in place. Fire clay was sufficiently refractory for this, but if in removing the door some of the luting fell in, it melted at once to an inconveniently thin slag, one from which silicon could be reduced into the metal.

When the stock of alundum ran low, attempts were made to substitute ordinary dead burned magnesite bonded with water-glass, but with very poor results. Plenty of alundum was almost as essential to the making of these experimental heats of steel as the charge itself; nevertheless, after the alundum lining had been in use some time and small globules of steel held in rough spots had become so oxidized between heats that some iron oxide was present, the lining would soften somewhat, and when the highest temperatures were being used to add large amounts of refractory ferro-alloys to very hot steel at the end of the heat, some of the lining would actually fuse and form a thickly fluid slag on the steel.

Had the furnace been used as a refining furnace, with refining slags, the lining would probably have given much more trouble.

## RECOMMENDED CHANGES IN FURNACE DESIGN.

Although the furnace as built operated satisfactorily, it became evident that slight changes in the design would give at once somewhat increased capacity and better refractory life. These changes principally involve increasing the shell diameter to 32½ inches and the shell length to 32 inches. With the same number of lining courses this would allow the hearth to be increased 3 inches in diameter and 2 inches in length. This would bring the arc farther from the walls and undoubtedly tend toward better refractory performance. The power required by this altered design would not differ greatly from that actually used.

If continuous operation of the furnace was desired, it would be better to increase still further the shell dimensions in order to provide for a single outer course of heat insulating brick, such as

Sil-o-cel or Armstrong Cork. This provision would cut down the radiation heat losses which would be met and tend to lower the power demands of the furnace on continuous operation.

## OPERATION OF INDIRECT-ARC FURNACE.

The procedure on a typical heat in the indirect-arc furnace was as follows: After the furnace was thoroughly cleaned out from the previous run, it was patched and dried, any large patch being in the lowest part of the furnace ready for preheating. The charge was then weighed out and both the ladle and the hot-top or "dozzler" preheated. The coated, dried, and smoked mold was prepared for use and placed where the waste heat from the ladle heater would heat it sufficiently to dry out any moisture. Tools, such as skimmers and stirrers, made from Armco iron so that if they melted in there would be no contamination, were cleaned and made ready, the furnace was luted up, and the arc started.

This furnace took a current of 500 to 750 amperes at 60 to 90 volts under load, had a power factor of about 90, and was run at 40 to 50 kilowatts, the rate of power input depending on the size of the charge.

Upon starting with a cold furnace the arc is unsteady, breaking from time to time for about 20 minutes. If the electrodes are not rounded off on the tips with reasonable smoothness, or if the electrodes do not enter the furnace on exactly the same center line, the period of unsteadiness may be 10 minutes longer. Any leak in the furnace at door or electrode holes will also accentuate the unsteadiness while it lasts. During this period the power input ranges from zero for the instant between the breaking of the arc to its restarting by the operator, to about 65 kilowatts the instant the arc is made, the average power input for the unsteady period being 30 to 35 kilowatts. After the furnace has reached a certain critical temperature the arc holds steadily, its length can be adjusted, and the power input thereby controlled.

### CHARGING FURNACE.

After heating for about an hour, using 40 to 45 kilowatt hours, the originally cold furnace is approximately up to running temperature when lined up without any carborundum brick and with the hearth of suitable size for a 50-pound charge. With the carborundum lining and the hearth large enough to hold 100 pounds the preheating of the cold furnace takes about one and one-quarter hours, using 60 kilowatt hours. The arc is broken, the door is taken out, the furnace drained and scraped free from any metal left in from the previous heat and from any slag soft enough to flow or to be scraped out, and the charge put in, taking care to pile it so that it does not touch the electrodes and is not likely to do so as it melts. All the iron, washed metal, any alloyed scrap such as ingot crops or "over metal" left after pouring the ingot (this scrap having, of course, been analyzed), the nickel, ferrotungsten, ferromolybdenum, ferrochromium, or other alloy called for which does not suffer loss when charged at the start, together with part of the ferromanganese and ferrosilicon, are charged. Since manganese and silicon might both be volatilized if placed directly under the arc, these are placed at the ends of the furnace, and preferably charged in large lumps rather than in crushed or powdered form.

The door is then replaced and luted in and the arc again started. Ten minutes is the average time from the breaking of the arc on the preheat to starting it again.

The furnace is then run at about 40 to 50 kilowatts, depending on the weight of the charges, until a calculated kilowatt hour's input has been reached. This depends on the kilowatt hours used in the preheat, on the time the door is open in charging, on the rate of power input, that is, whether the furnace is run at 40 or at 50 kilowatts, on the weight of the charge, and on the temperature desired for the steel at the end, which in turn depends on the amount of ferro-alloy to be added at the end and the ease with which it is taken up. For example, on a steel requiring only half a pound of readily absorbed ferro-alloys such as FeV, FeB, FeSi, or FeMn, 40 kilowatt hours on the heat proper after the preheat of the cold furnace will heat 50 pounds enough. On one requiring, say, 3 pounds of FeZr, FeU, or FeTi, 45 or even 47½ kilowatt hours may be used. The higher figures will usually result in the melting down of some and the spalling of some of the lining and the formation of a thickly fluid or crusty slag over the metal.

### ADDITION OF FERRO-ALLOYS.

When the scheduled kilowatt hour input has been reached, the heated ladle is set in the shank, the furnace opened, the electrodes taken out to give room to skim the metal, all slag or floating chunks of fallen lining skimmed off, the crushed or powdered ferro-alloys required, or if none are required save FeSi and FeMn, one-fourth to one-half pound of either of these, or a mixture of them according to what the charge calls for, is placed on the clean metal with a shovel, and at once thoroughly stirred in with an Armco iron rod. This portion of FeSi and FeMn is reserved until the end because such an addition appears to aid either in deoxidation or in "swamping" the dissolved gas and to be a desirable step in the production of sound ingots.

On the other hand this precaution may not be necessary, since in heat 1066 (Table 2, p. 28) the FeSi and FeMn reserved for the final addition were left out by mistake and the ingot was sound. If the steel was not hot enough or the amount of added ferro is more than can be taken up without chilling the steel too much, one or two of the small Thermit "Little Devil" cans for heating up dull iron or steel is plunged in on the end of an iron rod, the metal stirred with rod as soon as the reaction is over, the metal again hastily skimmed, the ladle put in place below the spout, and the charge poured.

Thermit cans are used only when the amount of ferro-alloy to be added is so great and so difficultly taken up that to heat the steel sufficiently to take up the ferro-alloy would ruin the lining or at least come dangerously near ruining it. They were required only on some of the heats in which large amounts of ferrouranium or ferrozirconium were used.

### DEOXIDIZING AGENT.

Unless the steel contains a very active deoxidizer, such as vanadium, uranium, or cerium, 0.01 to 0.005 per cent aluminum is thrown into the ladle just before the steel is poured. This is of course a larger percentage of aluminum than is used in commercial practice on electric furnace steel poured from large ladles, but amounts of this order of magnitude appear to be generally used as a final addition in crucible steel practice, as well as when pouring electric furnace steel from small hand ladles in foundries making electric steel castings. Moore[12] gives, however, a typical electric furnace charge as 0.025 per cent aluminum. The arsenal allowed 0.05 per cent Al in their specifications, and since this was the case no extended investigations were made to see if the use of Al could be avoided.

Since the stream of metal comes into contact with the air twice, once in pouring from furnace to ladle and once from ladle to mold, and the surface of the small stream necessary in handling a 50 or 100 pound charge is greater in proportion to the weight of metal than is the case of large heats, the use of Al was considered desirable, and was the regular practice unless other strong deoxidizers were present or unless aluminum was carried in by ferro-alloys made by reduction with aluminum. If it had been possible to heat the ladle hotter and then allow more time for degasification in the ladle before pouring into the mold, the use of aluminum could probably have been avoided.

### POURING METAL.

The ladle was normally allowed to stand until the metal was absolutely quiet and until it had cooled to the point where it would either pour clean or leave only a thin skull in the ladle. The best pouring temperature seemed to be about 1,600° C., determined by an optical pyrometer. The 1,600° C. figure is corrected[13] for deviation from black body temperature, the observed temperature being 1,470° C. Of course in some tests the addition of large amounts of ferro-alloy chilled the metal so that it had to be poured at once into the mold and even then might leave a rather heavy skull, but usually it was possible to hold the metal in the ladle from one to two minutes before it was poured into the mold.

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[12] Moore, W. E., Electric furnaces for steel foundries: Elect. Jour., vol. 16, 1919, p. 365.

In pouring, care was taken to skim back any slag, oxide skin, or other floating impurities, to pour steadily, and to keep the stream from hitting the sides of the mold. When the mold had filled within about an inch of the top, pouring was interrupted, the hot-top brick inserted and held from floating up, and pouring again started slowly until the metal had frozen about the inserted part of the hot-top brick and then the hot-top filled to the top. The hot-tops were 6½ inches tall, with a 2 inch diameter hole. The ingot molds finally used were of the usual split form, the ingot being about 22 inches long, 2½ by 2½ inches near the butt (rounding off below this) and 3 by 3 inches at the top. In the second series of zirconium steels, 6 by 3 inch molds were used, with the usual hot-tops for that size. Unless the hot-top was heated hotter than could be done with the apparatus at hand, however, it was not sufficient, as the ingots piped below the hot-top. The cropped ingots gave more metal below the pipe when no hot-top was used on this size, than when one was used that was too cool.

The 3 by 3 inch mold plus the extension made by the hot-top held about 44 to 47 pounds of steel, hence the regular charge was made up to a total of 46½ to 50 pounds when one of these molds was used.

All the steels made piped very deeply when no hot-top was used, but by using one and cropping the 3 by 3 inch ingot about 1 inch below the bottom of the hot-top, the pipe was all removed and sound metal reached. After cropping about 38 to 40 pounds of sound metal was left. In many cases the bottom of the ingot was drilled for analysis for the study of possible segregation between the bottom and the top, the other sample being taken by drilling just below the pipe. In these cases the balance of the ingot, ready for forging or rolling, was reduced to 35 to 37 pounds.

After the hot-top had been filled the top was kept open by poking it with an iron rod to prevent bridging over with frozen metal and to allow the escape of any gas released while freezing. Just as soon as the metal had set the ingot was taken from the mold, the piped portion that was inside the hot-top was knocked off and the ingot at once buried in fine lime, for slow cooling, in order to soften it for drilling the sample and for cropping. It usually took 36 hours for the ingot to cool enough to be handled.

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[13] Burgess, G. K., Temperature measurements in Bessemer and open hearth practice: Bureau of Standards, Tech. Paper 91, 1917, pp. 9, 16.

While the ingot was being poured the furnace operator poked out the electrode holes, scraped the furnace free from all plastic slag, and drained it from any steel that had remained in pockets in the lining, charged the next heat, replaced the electrodes, having jarred them to shake off any loosely adhering graphite powder that may be on them, replaced and luted the door, and started the second heat.

When the furnace is hot from the first run, of course no preheating is required. The arc may fluctuate a trifle for, say, five minutes, but seldom breaks, and has soon settled down so as to require no attention.

## RUNNING ADDITIONAL HEATS.

The power input required on the second heat is reduced from the 40 to 47½ kilowatt hours required on the run proper of the first heat to from 30 to 38 kilowatt hours on a 50-pound charge, and, were it wise to continue taking off heats without patching the lining, the power required would continue to fall, as the furnace does not appear to have reached a thermal equilibrium through its walls even at the end of the second heat.

In calculating a charge, one must allow for losses of C, Si, and Mn, as well as of the still more readily oxidizable elements such as U and Zr. The loss of Si is a fairly constant factor and no very great difficulty is found in coming reasonably close to the desired Si content. If the charge comes in contact with fire clay or fire brick, it may reduce Si therefrom, with a loss of C or Mn, and the Si in the steel may rise even above the amount charged. Barring such a case, which may occur if the lining becomes very badly damaged in the course of a heat so that the steel gets back to the fire-brick layer of the lining, the Si in the steel is lower than that charged. Mn always shows a loss, and a rather variable one, but it can usually be predicted with a fair degree of accuracy.

## CONTROL OF CARBON.

Carbon is still the hardest variable to control, the greatest danger being with a bulky charge, which may have to be piled higher than the electrodes in some places and as the charge melts it may fall down and part of it rest against an electrode, thus absorbing carbon. Another source of carbon acquisition is the powdering or flaking off of the electrodes, which may occur to a variable extent, thus dropping a variable amount of graphite into the bath.

It is fairly easy to bring out duplicate heats, of which, say, 80 per cent will be within a range of 10 points of carbon. If the charge is made up to calculate 0.50 per cent C, the steels will run mostly between 0.35 and 0.45 per cent C. Backert[14] gives 0.30 to 0.40 as the range that must be allowed in crucible steel when 0.35 is desired. If it is calculated for 0.30, they will run mostly between 0.25 and 0.30 per cent, while if calculated for 0.15 or below, what few steels have been made with this low carbon content persist in coming 0.15 to 0.20 per cent. This shows the effect of the graphite that drops off the electrodes, the adventitious carbon from this source more than making up for that lost by oxidation in the latter case, almost balancing it in the case of steels calculated for 0.30 per cent C but being overbalanced by the oxidation loss on those calculated for 0.50 per cent C.

When the furnace is enlarged to a capacity of 100 pounds, the adventitious graphite from the electrodes plays a less important rôle and the carbon content of the product can be more closely predicted.

Although the lack of complete certainty of carbon control is a disadvantage, it is perhaps no worse in the experimental electric furnace than in crucible practice. Figures from the Watertown Arsenal show that in making up some experimental steels in their crucible-steel foundry in about 100-pound heats, the following figures for carbon were obtained:

*Per cent of carbon aimed for and obtained in 17 heats in crucible furnaces.*

| Per cent of C aimed for. | Per cent of C obtained. | Per cent of C aimed for. | Per cent of C obtained. |
|---|---|---|---|
| 0.40 | 0.46 | 0.30 | 0.20 |
| .40 | .49 | .30 | .20 |
| .40 | .29 | .30 | .36 |
| .30 | .38 | .30 | .42 |
| .50 | .55 | .30 | .45 |
| .40 | .44 | .30 | .35 |
| .40 | .43 | .30 | .32 |
| .40 | .38 | .40 | .48 |
| .30 | .26 | | |

This shows a range of —11 to +15 points from that aimed at.

In one series in particular, in which the specifications were C, 0.40 to 0.50 per cent; Si, 0.15 to 0.35 per cent; Mn, 0.35 to 0.80 per cent; and W, 1 to 15 per cent in 1 per cent steps, the results obtained in the indirect-arc furnace are shown in Table 2.

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[14] Backert, A. O., A. B. C. of iron and steel, 1915, p. 117.

TABLE 2.—*Results of series of heats in indirect-arc furnace.*

| Number of heats. | Per cent calculated. | | | | Per cent obtained. | | | | Per cent of change. | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | C. | Si. | Mn. | W. | C. | Si. | Mn. | W. | C. | Si. | Mn. | W. |
| 1058 | 0.55 | 0.38 | 0.07 | 1.8 | 0.49 | 0.26 | 0.54 | 1.70 | −0.06 | −0.12 | −0.33 | −0.10 |
| 1059 | .57 | .40 | .90 | 2.5 | .49 | .32 | .59 | 2.20 | − .08 | − .08 | − .31 | + .30 |
| 1060 | .54 | .38 | .87 | 1.2 | .48 | .20 | .60 | 1.15 | − .06 | − .18 | − .27 | − .05 |
| 1061 | .54 | .37 | .87 | 3.1 | .41 | .24 | .61 | 3.05 | − .13 | − .13 | − .26 | − .25 |
| 1062 | .54 | .37 | .87 | 4.2 | .41 | .28 | .51 | 3.95 | − .13 | − .05 | − .36 | − .25 |
| 1063 | .54 | .37 | .87 | 5.3 | .42 | .27 | .58 | 4.70 | − .12 | − .10 | − .29 | − .60 |
| 1064 | .54 | .39 | .88 | 6.15 | .49 | .21 | .62 | 6.25 | − .05 | − .18 | − .28 | + .10 |
| 1065 | .56 | .39 | .88 | 7.15 | .47 | .20 | .68 | 7.35 | − .09 | − .19 | − .20 | + .20 |
| 1066 | .54 | .26 | .57 | 8.2 | .42 | .13 | .40 | 8.45 | − .12 | − .13 | − .17 | + .25 |
| 1067 | .54 | .38 | .83 | 9.25 | .45 | .19 | .63 | 9.70 | − .09 | − .19 | − .20 | + .45 |
| 1068 | .54 | .38 | .84 | 8.15 | .42 | .23 | .64 | 8.50 | − .12 | − .15 | − .20 | + .35 |
| 1069 | .54 | .37 | .84 | 10.0 | .42 | .20 | .62 | 10.00 | − .12 | − .17 | − .20 | ± .00 |
| 1070 | .54 | .35 | .76 | 11.1 | .38 | .33 | .50 | 11.00 | − .16 | − .02 | − .26 | − .10 |
| 1071 | .54 | .38 | .84 | 12.0 | .40 | .29 | .53 | 11.80 | − .14 | − .11 | − .31 | − .20 |
| 1072 | .54 | .39 | .84 | 13.1 | .37 | .27 | .63 | 12.80 | − .17 | − .12 | − .21 | − .30 |
| 1073 | .54 | .40 | .85 | 14.1 | .46 | .30 | .65 | 13.80 | − .08 | − .10 | − .20 | − .30 |
| 1074 | .54 | .38 | .84 | 15.1 | .42 | .24 | .73 | 15.20 | − .12 | − .14 | − .11 | + .10 |
| 1075 | .54 | .39 | .84 | 7.05 | .42 | .25 | .74 | 7.15 | − .12 | − .14 | − .10 | + .10 |
| Average | | | | | | | | | − .10 | − .13 | − .23 | ± .0 |

At the beginning of the series, 0.05 per cent Al was used in the ladle and cut to 0.03 per cent by the middle of it. On later runs it was cut to 0.01 to 0.005 per cent. The Armco iron used contained 0.02 per cent S, and 0.004 per cent P, the washed metal below 0.02 per cent each of S and P, while the FeW contained 85 per cent W, 0.9 per cent Si, 0.13 per cent C, 0.05 per cent P, 0.01 per cent S. The FeSi was ordinary 50 per cent Si ferro, and the FeMn ordinary 80 per cent Mn ferro with 6 per cent C and about 0.25 per cent P. Small amounts of skeleton scrap steel from the punch presses of a local airplane factory containing 0.1 per cent C, 0.45 per cent Mn, and below 0.04 per cent each of S and P were also used, and crop ends of ingots were remelted in most of the heats. Since the S could not run over 0.03 or the P over 0.02 in the finished steels, no analysis was made for these elements. Analysis for W was made only for routine control, no correction being made for iron oxide carried down with the $WO_3$, nor were special precautions taken for the precipitation of the last traces of W. The average results indicate that the tungsten, which was charged at the start of the heat, was quantitatively recovered. According to Tyler[15], in British crucible tool-steel melting, 20 per cent tungsten powder has to be added to give 18 per cent W in the steel. The powder is stated to contain 98 to 98 per cent W.

In one series of high-silicon steels (2 per cent Si or more), with 65 to 70 pound heats with and without zirconium, the charge being calculated to 0.52 per cent C and 0.36 to 0.43 per cent C being aimed at, 26 out of 34 steels were in that range, 1 was 0.35 per cent, and 7 from 0.46 to 0.5 per cent C.

In another series of 34 plain, molybdenum, vanadium, and cerium steels, in which steels of 0.38 to 0.41 per cent C were desired, 17 were obtained in that range, 5 from 0.35 to 0.37 per cent C and 6 from 0.42 to 0.44 per cent C and one each of 0.45, 0.46, 0.47, 0.50, 0.53, and 0.54 per cent C. The charges weighed 75 to 95 pounds and were calculated for 0.46 per cent C. The percentage loss of carbon is slightly lower as the size of the heat is increased.

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[15] Tyler, P. M., High-speed steel manufacture in Sheffield: Iron Age, vol. 107, 1921, p. 370.

Although less uniform results than the above were obtained in some melts, these series were the only long ones made in which the operator was not continually faced with new variables in having to add different ferro-alloys of unknown behavior, and this series probably shows the capabilities of the furnace fairly. It would appear that the indirect-arc electric furnace is at least as likely to give the carbon composition desired on an experimental heat as is the crucible furnace.

## SILICON AND MANGANESE STEELS.

Table 2 shows the loss of Si and Mn on steels of fairly normal Si and Mn content for castings and forgings. The losses when steels of higher Si and Mn content are aimed at are shown below, the figures being taken at random from a larger number.

*Loss of silicon and manganese on steels of high Si and Mn content.*

| No. of heat. | Per cent calculated. | | Per cent found. | | Difference between per cent calculated and found. | |
|---|---|---|---|---|---|---|
| | Si. | Mn. | Si. | Mn. | Si. | Mn. |
| 1102 | 1.41 | 0.80 | 1.15 | 0.65 | 0.26 | 0.15 |
| 1104 | .72 | .82 | .66 | .58 | .06 | .24 |
| 1113 | 1.42 | .89 | 1.17 | .76 | .25 | .13 |
| 1114 | .71 | .89 | .52 | .65 | .19 | .14 |
| 1120 | 1.35 | 1.23 | 1.00 | .97 | .35 | .26 |
| 1128 | 1.49 | .95 | 1.08 | .87 | .41 | .08 |
| 1135 | 1.50 | .90 | 1.44 | .83 | .06 | .07 |
| 1136 | 2.20 | .90 | 1.78 | .83 | .32 | .07 |
| 1147 | 1.55 | .91 | 1.04 | .81 | .41 | .10 |
| 1163 | 1.55 | .90 | 1.38 | .81 | .17 | .09 |
| 1164 | 1.55 | .90 | 1.48 | .80 | .07 | .10 |
| 1165 | 1.55 | .90 | 1.48 | .76 | .07 | .14 |
| 1166 | 1.55 | .90 | 1.33 | .80 | .22 | .10 |
| 1167 | 1.55 | .90 | 1.26 | .75 | .29 | .15 |
| 1168 | 1.55 | .90 | 1.36 | .77 | .19 | .13 |
| 1169 | 1.55 | .90 | 1.33 | .82 | .22 | .08 |
| 1170 | 1.55 | .90 | 1.27 | .79 | .28 | .11 |
| 1171 | 1.55 | .90 | 1.34 | .78 | .21 | .12 |
| 1172 | 1.55 | .90 | 1.32 | .76 | .23 | .14 |
| 1173 | 1.55 | .90 | 1.36 | .79 | .19 | .11 |
| 1198 | 1.43 | .92 | 1.08 | .71 | .35 | .21 |
| 1199 | 1.44 | .92 | 1.12 | .75 | .32 | .17 |
| 1200 | 1.45 | .93 | 1.18 | .74 | .27 | .19 |
| 1201 | 1.45 | .93 | 1.26 | .84 | .19 | .09 |
| 1206 | 1.40 | .95 | 1.25 | .80 | .15 | .15 |
| 1207 | 1.40 | .95 | 1.30 | .80 | .10 | .15 |
| 1208 | 1.40 | 1.30 | 1.25 | 1.00 | .15 | .30 |
| 1209 | 1.40 | 1.30 | 1.20 | 1.15 | .20 | .15 |
| 1214 | 1.82 | .95 | 1.40 | .80 | .42 | .15 |
| 1226 | 1.91 | 1.00 | 1.60 | .90 | .31 | .10 |
| 1227 | 1.91 | 1.00 | 1.45 | .85 | .46 | .15 |
| 1236 | 1.50 | 1.50 | 1.45 | 1.10 | .05 | .35 |
| 1237 | 2.31 | 1.01 | 2.18 | .94 | .13 | .07 |
| 1238 | 1.40 | 2.18 | 1.25 | 2.00 | .15 | .18 |
| 1251 | 1.66 | 1.89 | 1.25 | 1.46 | .31 | .43 |
| 1300 | 2.50 | .87 | 2.31 | .83 | .19 | .04 |
| 1303 | 2.50 | .87 | 2.33 | .81 | .17 | .06 |
| 1325 | 2.50 | .87 | 2.47 | .70 | .03 | .17 |
| Average | ...... | ...... | ...... | ...... | .20 | .15 |

The greatest deviations from the average loss are: Si, + 0.15 and —0.27 per cent; Mn, + 0.07 and —0.28 per cent.

About 75 per cent of these steels come within limits of 0.10 per cent Si either side of the average loss of Si, and about 90 per cent of them come within limits of 0.10 per cent Mn either side of the average loss of Mn.

In the steels listed above, all the Si and Mn were added at the start save about 100 grams each of FeSi and FeMn added at the end to help "swamp" occluded gases. Slightly different results are obtained when most of the Si for a high Si steel is added at the end of the heat as SiZr alloy. Only the difference between the total to be added and that carried by the SiZr is put in at the start, all the Mn save about 50 grams of FeMn, which is added at the end to help control gases, being charged at the start. Under these conditions, the bulk of the work of deoxidation during the melting of the steel falls on the Mn, the loss of Si being less and that of Mn greater, as the following figures show:

*Per cent of silicon and manganese calculated and found in 18 heats.*

| No. of heat. | Per cent calculated. | | Per cent found. | | Difference between per cent calculated and found. | |
|---|---|---|---|---|---|---|
| | Si. | Mn. | Si. | Mn. | Si. | Mn. |
| 1180 | 1.60 | 0.95 | 1.55 | 0.63 | 0.05 | 0.3 |
| 1182 | 1.60 | .95 | 1.55 | .76 | .05 | .1 |
| 1184 | 1.50 | .90 | 1.20 | .61 | .20 | .2 |
| 1185 | 1.50 | .90 | 1.37 | .71 | .13 | .1 |
| 1186 | 1.50 | .90 | 1.37 | .74 | .13 | .1 |
| 1187 | 1.50 | .90 | 1.40 | .67 | .10 | .2 |
| 1188 | 1.50 | .90 | 1.38 | .74 | .12 | .1 |
| 1189 | 1.50 | .90 | 1.50 | .73 | .00 | .1 |
| 1191 | 1.45 | .90 | 1.40 | .77 | .05 | .1 |
| 1192 | 1.46 | .90 | 1.45 | .75 | .01 | .1 |
| 1193 | 1.46 | .90 | 1.45 | .67 | .01 | .2 |
| 1194 | 1.45 | .90 | 1.42 | .55 | .03 | .2 |
| 1195 | 1.45 | .90 | 1.52 | .65 | + .07 | .2 |
| 1196 | 1.46 | .90 | 1.50 | .65 | + .04 | .2 |
| 1197 | 1.46 | .90 | 1.50 | .60 | + .04 | .3 |
| 1212 | .90 | .95 | .80 | .70 | .10 | .2 |
| 1213 | .90 | .95 | .80 | .65 | .10 | .3 |
| 1289 | 1.65 | 1.13 | 1.57 | .83 | .08 | .3 |
| Average | ........ | ........ | ........ | ........ | .06 | .2 |

No difference was found in the action of ordinary 80 per cent Mn 6 per cent C ferromanganese and 50 per cent ferrosilicon as agains siliconmanganese alloys of 75 per cent Mn, 6 per cent Si, 4.3 per cen C; 73 per cent Mn, 10 per cent Si, and 3 per cent C; or 72.5 per cen Mn, 16.5 per cent Si, and 1.5 per cent C made in another investigation.[16]

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[16] Gillett, H. W., and Williams, C. E., Electric smelting of domestic manganese ores Bull. 173, Bureau of Mines, 1920, chap. 10, pp. 151–186.

## MOLYBDENUM STEELS.

With ferromolybdenum of 50 per cent Mo, 2.2 per cent C, 1.2 per cent Si, 0.12 per cent Mn, 0.57 per cent S, 0.18 per cent P, or 42.4 per cent Mo, 0.08 per cent C, 7.2 per cent Si, 0.2 per cent S, added at the start of the heat, the recovery of Mo was practically quantitative, as is shown below:

*Per cent of molybdenum calculated and found.*

| No. of heat. | Per cent of Mo calculated. | Per cent of Mo found. | No. of heat. | Per cent of Mo calculated. | Per cent of Mo found. |
|---|---|---|---|---|---|
| 1097 | 3.02 | 3.00 | 1358 | .75 | .75 |
| 1092 | 2.25 | 2.05 | 1360 | .75 | .73 |
| 1089 | 2.21 | 1.90 | 1366 | .75 | .75 |
| 1096 | 2.09 | 1.90 | 1369 | .75 | .76 |
| 1088 | 1.23 | 1.15 | 1337 | .35 | .37 |
| 1091 | 1.10 | 1.05 | 1342 | .35 | .34 |
| 1135 | .81 | .78 | 1345 | .35 | .36 |
| 1136 | .81 | .77 | 1350 | .35 | .31 |
| 1336 [a] | .75 | .67 | 1355 | .35 | .35 |
| 1341 | .75 | .73 | 1361 | .35 | .35 |
| 1344 | .75 | .68 | 1367 | .35 | .34 |
| 1349 | .75 | .83 | 1371 | .35 | .37 |

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[a] Mo analyses on Nos. 1336 to 1371 by Vanadium Corporation of America.

## CHROMIUM STEELS.

Using low-carbon ferrochrome of about 65 per cent Cr, and 0.40 per cent C, added at the start, the following recoveries were obtained:

*Recoveries of chromium calculated and found.*

| No. of heat. | Per cent of Cr calculated. | Per cent of Cr found. | No. of heat. | Per cent of Cr calculated. | Per cent of Cr found. |
|---|---|---|---|---|---|
| 1124 | 1.02 | 0.96 | 1355 | .92 | .88 |
| 1125 | 2.03 | 1.99 | 1356 | .92 | .84 |
| 1126 | 3.5 | 3.61 | 1357 | .92 | .95 |
| 1127 | 3 | 2.77 | 1358 | .92 | .86 |
| 1177 | 2 | 1.95 | 1364 | .92 | .83 |
| 1178 | 2 | 1.98 | 1366 | .92 | .78 |
| 1343 [a] | 1 | .94 | 1367 | .92 | .83 |
| 1344 | 1 | .95 | 1368 | .92 | .82 |
| 1345 | 1 | .89 | 1369 | .92 | .79 |
| 1346 | 1 | .93 | 1370 | .92 | .92 |
| 1347 | 1 | .98 | 1348 | .75 | .65 |
| 1359 | 1 | .92 | 1349 | .75 | .67 |
| 1360 | 1 | .93 | 1350 | .75 | .68 |
| 1361 | 1 | .84 | 1351 | .75 | .74 |
| 1362 | 1 | .97 | 1365 | .75 | .77 |
| 1353 | .92 | .83 | 1374 | .75 | .84 |

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[a] Cr analyses on Nos. 1343 to 1374 by Vanadium Corporation of America.

## VANADIUM STEELS.

Using ferrovanadium of 36 per cent V, 0.26 per cent C, 2.3 per cent Si, 0.13 per cent P, 0.15 per cent Mn, 0.13 per cent Cu, S trace, the following results were obtained:

*Recoveries of vanadium calculated and found.*

| No. of heat. | Per cent V— | | When ferro added. | Size of ferro. |
|---|---|---|---|---|
| | Calculated. | Found. | | |
| 1121 | 1.03 | 0.98 | At start | Hickory nut. |
| 1122 | 5.38 | 3.00 | Near end [a] | Do. |
| 1123 | 2.22 | 1.95 | .....do. [a] | Do. |
| 1173 | .26 | .29 | At end | Pea and smaller. |
| 1207 | .31 | .32 | .....do | Do. |
| 1273 | .35 | .33 | .....do | Do. |
| 1346 [b] | .25 | .20 | .....do | Do. |
| 1351 | .25 | .24 | .....do | Do. |
| 1356 | .25 | .20 | .....do | Do. |
| 1362 | .25 | .24 | .....do | Do. |
| 1368 | .25 | .19 | .....do | Do. |

[a] Furnace opened after rest of charge was melted, FeV added, furnace closed and run 5 to 10 kilowatt hours more.

[b] Analyses 1346 to 1368 by Vanadium Corporation of America.

Up to 1 per cent there appeared to be a practically quantitative recovery of V, but on heat 1223 there was a notable loss and on 1222 a great one. In these heats a slag or scum formed continually on the steel as it was held in the ladle for degasification, the scum forming again as fast as it was skimmed off.

## NICKEL STEELS.

The recovery of nickel added at the start with the rest of the charge is quantitative, as is shown by the following representative figures.

*Per cent of nickel calculated and found.*

| No. of heat. | Per cent of Ni— | | | | No. of heat. | Per cent of Ni— | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Calculated. | Found. | | | | Calculated. | Found. | | |
| | | Top. | Butt. | Average. | | | Top. | Butt. | Average. |
| 1299 | 3 | 2.96 | 2.96 | 2.96 | 1308 | 3 | 2.96 | 2.98 | 2.97 |
| 1300 | 3 | 2.94 | 2.96 | 2.95 | 1309 | 3 | 2.96 | 2.92 | 2.94 |
| 1302 | 3 | 3.01 | 3.04 | 3.02 | 1310 | 3 | 2.96 | 2.90 | 2.93 |
| 1303 | 3 | 3.02 | 3.00 | 3.01 | 1322 | 3 | 3.03 | 2.97 | 3.00 |
| 1304 | 3 | 2.91 | 2.95 | 2.92 | 1323 | 3 | 2.97 | 2.90 | 2.94 |
| 1305 | 3 | 3.03 | 3.05 | 3.04 | 1324 | 3 | 2.95 | 2.97 | 2.96 |
| 1306 | 3 | 3.00 | 3.00 | 3.00 | 1325 | 3 | 3.02 | 2.97 | 3.00 |
| 1307 | 3 | 2.98 | 2.98 | 2.98 | | | | | |

## COPPER-NICKEL STEELS.[18]

Copper added at the start with the rest of the charge, in steels carrying nickel also, and from 0.8 to 1 per cent Mn gave the following:

*Per cent of copper and nickel charged and found in copper-nickel steels.*

| No. of heat. | Charged. | | Found by analysis. | | | |
|---|---|---|---|---|---|---|
| | Ni, per cent. | Cu, per cent. | Ni, per cent. | Per cent Cu. | | |
| | | | | Top. | Butt. | Average. |
| 1279 | 2.43 | 0.6 | 2.46 | 0.63 | 0.60 | 0.61 |
| 1280 | 2.43 | .6 | 2.45 | .58 | .53 | .55 |
| 1283 | 2.60 | .4 | 2.60 | .35 | .37 | .36 |
| 1284 | 2.60 | .6 | 2.58 | .56 | .54 | .55 |
| 1285 | 2.43 | .6 | 2.53 | .61 | .63 | .62 |
| 1286 | 2.43 | .6 | 2.56 | .65 | .63 | .64 |

## ALUMINUM STEELS.

The recovery of aluminum was fairly high, as is shown by the following:

*Recovery of aluminum.*

| No. of heat. | Per cent Al— | | |
|---|---|---|---|
| | Charged. | Found. | Lost. |
| 1090 | 5.25 | 4.90 | 0.35 |
| 1147 | .38 | .30 | .08 |
| 1206 | .43 | .23 | .20 |
| 1270 | .60 | .45 | .15 |
| 1329 | .40 | .20 | .20 |

In these runs, the aluminum was added at the end of the heat, being placed on the metal in the furnace just before pouring, and thoroughly stirred in. In heat 1090 and several others in which 5 per cent and 2 per cent Al were added, but on which no analysis was made, as the exact percentage of aluminum was not important, an oxide skin continually formed in the ladle, and in pouring a skin tended to form about the stream of metal. The metal seemed to hold some oxide on the heats highest in aluminum, and acted slushy in pouring, even though it was fully as hot as usual.

## URANIUM STEELS.

The data on the production of uranium steels in the indirect-arc furnace are shown in Table 3.

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[18] Compare Clamer, G. H., Cupronickel steel: Metal Ind., vol. 8, 1910, p. 303.
Escard, J., Les ferro-alliages complexes: Jour. du four Electrique, vol. 29, 1920, p. 36.
Metal Industry (London), Copper in steel, vol. 16, 1920, p. 435.

### TABLE 3.—*Production of uranium*

| No. of heat. | Total weight of charge. | FeU added, analysis. | | | | | Total kw.h. used.[a] | 1st or 2d heat. | FeU added at— | | Size FeU. | Per cent of U. | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Lbs. | No. of sample. | U. | C. | Si. | | | Kw. h. | Start or end. | | Calculated. | Analysis. | |
| | | | | | | | | | | | | | Top. | Butt. |
| | *Lbs.* | | | *P. ct.* | *P. ct.* | *P. ct.* | | | | | | | | |
| 1049 | 43 | 2<br>1 | [b] 78<br>97 | 46<br>43 | 2.1<br>2.15 | 1.8<br>.7 | 40 | 1st.. | 40 | End | Pea and smaller. | 3.2 | 0.75 | ...... |
| 1051 | 39 | 2.5 | 78 | 46 | 2.1 | 1.8 | 45 | 1st.. | 45 | End | Hickory nut and smaller.[c] | 3+<br>.2 | 1.09 | ...... |
| 1052 | 38.5 | 2.5<br>1 | 95<br>74 | 58.5<br>60 | 3.65<br>2.4 | 1.2<br>2.4 | [c]45 | 2d... | 45 | End | .....do....... | [c]5.4+<br>.1 | .18 | ...... |
| 1053 | 39.5 | 2<br>2 | 47<br>74 | ..... | ...... | ..... | 41 | 1st.. | 41 | End | Pea........... | 5.85 | [c]2.5 | ...... |
| 1054 | 39.5 | 3.8<br>.8 | 85<br>72 B | 64<br>61 | 3.25<br>1.7 | .4<br>.3 | [c]39 | 2d... | 39 | End | .....do........ | 7.45 | [c]3.65 | ...... |
| 1056 | 39.3 | 6 | 75 | 51.5 | 2.05 | .4 | 45 | 1st.. | 45 | End | Hickorynut and smaller. | 7.78 | 7.75 | ...... |
| 1076 | 50 | 3.75 | 35 | 40 | 4.1 | 1.8 | 44 | 1st.. | 45 | End | Egg and smaller. | 3 | 1.92 | ...... |
| 1077 | 50 | 3.9<br>2 | 35<br>40 | 40<br>42.5 | 4.1<br>4.5 | 1.8<br>.3 | 40 | 1st.. | 0 | Start | .....do........ | 4.78 | 1.66 | 1.76 |
| 1078 | 50 | 1.4<br>2.8<br>2<br>1.1<br>3.5 | 40<br>97<br>71<br>64<br>87 | 42.5<br>43<br>36.5<br>35<br>65.5 | 4.5<br>2.15<br>.9<br>3.9<br>1.5 | .3<br>.7<br>4.7<br>1.4<br>.5 | 28 | 2d... | 0 | Start | Egg to pea and smaller. | 10.5 | 1.5 | 1.75 |
| 1079 | 49.5 | .5<br>.9<br>.6<br>.7<br>.5<br>2.4<br>5.5 | 33<br>40<br>43<br>50<br>78<br>83<br>88 | 39.5<br>42.5<br>49.5<br>65<br>46<br>56<br>59 | 4<br>4.5<br>5<br>5.3<br>2.1<br>1.7<br>1.3 | .4<br>.3<br>.6<br>.5<br>1.8<br>1.6<br>.6 | 12.5 | 1st.. | 0 | Start | .....do........ | 12.5 | 1 | 1.09 |
| 1080 | 50 | 1<br>1.4 | 86<br>41 | 68.5<br>45 | 5<br>4.2 | .3<br>.4 | 37.5 | 1st.. | 0 | Start | Butternut and smaller. | 4.6 | .3 | .35 |
| 1081 | 50 | 4.9 | 41 | 45 | 4.2 | .4 | 41 | 1st.. | 0 | Start | Hickory nut and smaller. | 4.42 | 1.53 | .16 |
| 1082 | 50 | 3.25<br>1.6<br>.2 | 80<br>75<br>74 | 67<br>51.5<br>60 | 1.1<br>2.05<br>2.4 | .9<br>2.5<br>2.4 | 47 | 1st.. | 41 | Near end. | ....do........ | 7.05 | .17 | .04 |
| 1083 | 50 | 1.5<br>1.7<br>.5 | 73<br>57<br>69 | 70<br>33<br>37.5 | 1.7<br>2.7<br>1.75 | 3.8<br>1.4<br>2 | 40 | 1st.. | 35 | Near end. | ....do........ | 4.4 | .05 | ...... |
| 1084 | 46 | 2<br>4 | 61<br>91 | 45<br>66 | 3.7<br>1.8 | .3<br>.8 | 40 | 1st.. | 0<br>40 | Start<br>End | Hickory.....<br>Pea.......... | [d]2.25<br>[e]5.75<br>8 | 1.89 | 2.32 |

[a] Preheat not included.

[b] See Gillett, H. W., and Mack, E. L. Preparation of Ferrouranium, Tech Paper 177, Bureau of Mines, 1917, for preparation of these samples of FeU.

[c] See note.

[d] Start.

[e] End.

*steels in indirect-arc furnace.*

Remarks.

Metal was too cold to take all FeU into solution. In forging, the first blow smashed the end of the ingot struck. Forging was probably attempted with metal too hot. Balance of ingot forged well at a lower temperature.

0.2 per cent U in charge at start—crop end from heat 1049; 3.0 per cent U in ferrouranium was added at end.

0.1 per cent U in crop end at start, 5.4 per cent in FeU at end. This was a second heat; that is, it was made after heat 1051, not after a preheat of the empty furnace. The metal was very hot. A little slag, such as had been used in the direct-arc furnace (75 gr. CaO, 20 gr. $SiO_2$, 5 gr. $CaF_2$) was used on heats 1049, 1051, and 1052, the first in the indirect-arc furnace, in the hope of protecting the steel from oxidation. On heat 1052, the slag was so thin all could not be skimmed off before the FeU was added as it was on the two previous heats. When the FeU was added through the slag and stirred in, the slag foamed up to a large volume, and was finally skimmed off. The metal was so hot that it was left to cool in the ladle about four minutes before it was poured and was, moreover, cooled by thrusting part way in, and then withdrawing a small ingot of "over metal" from the previous heat, which did not melt in, but did chill the metal. No slag formers were added on later heats.

Sample taken from crop gave 2.5 per cent U. Samples from sound metal of both ends of forged bar, after cropping ragged end off bar, gave 3.39 per cent and 3.57 per cent U.

Both ends of the bar shattered in forging so that only about 1 foot of bar (1 inch diameter) was obtained. Samples from the two ends of this bar gave 4.61 per cent U and 0.68 per cent U.

Metal was very hot, but addition of 6 pounds of cold ferro to 33.3 pounds of molten metal chilled it so that not all could be poured from the furnace and a large skull was left in the ladle, though the metal was poured at once. This bar smashed completely when attempt was made to forge it.

Metal was very hot, but large chunks of ferro (which were added to see if the previous additions of finer material had caused loss of U by oxidation of the surface of the ferro while charging) did not dissolve well and the metal was slushy, as though carrying undissolved particles, although it was very hot. The metal acted dead, and the ingot showed a deep pipe, but the ingot surface was full of holes.

Although the analysis shows a high loss of U, there was very little slag on the metal when the furnace was opened. The FeU was put in at the start with the rest of the charge.

FeU was charged at start in this heat also, but some of ferros 87 and 97 were in pieces covered with a coating of uranium oxide, having been spilled when the FeU was poured in preparing the ferros. Some of these pieces went through the steel furnace without melting, about 4 pounds of such material being scraped from the bottom of the steel furnace.

Out of 11 pounds of FeU charged at the start, about 4 pounds (samples 83 and 88) was spilled material covered with an oxide skin. A few chunks of FeU went through the steel furnace unfused. There was a good deal of loose dross on the metal, some pieces of lining having fallen off, and much uranium oxide was found in this dross after it was skimmed and cooled.

All the FeU charged at the start was good clean metal. Twelve and one-half pounds of heat 1056 ingot (calculated to contain 7.75 per cent U on basis of analysis of drillings from top) was also charged. About one-half pound of undissolved FeU was scraped from the bottom of the furnace after it had cooled, together with much black powder, which was mainly uranium oxide. The steel was not slushy.

All the FeU was clean. Metal was very hot, but acted as if it contained undissolved particles. No unfused FeU was found in the furnace after cooling, but a considerable amount of black powder, mostly uranium oxide, was found.

The ferrouranium (good clean metal) was added after the steel was fully molten; then the furnace was closed and heated 15 minutes (using 6 kw. h.) and rocked back and forth to mix the ferro with the steel during this 15 minutes. In addition to the FeU thus added near the end, there was added 23 pounds of crop and skull from heat 1076, calculated as containing 1.92 per cent U. The metal was very hot, poured well, and did not act slushy or pasty. The usual black powder was found in the furnace, but no unfused ferro.

Procedure was similar to that of preceding heat. Clean ferro was added after steel was melted and the furnace heated 8 minutes, with continual rocking, using 5 kw. h. Twenty and one-half pounds of heat 1076 was charged at the start also, and its U content was included in the calculation. Considerable lining fell during the heat, the floating material being skimmed before the ferrouranium was added. The metal was very hot and appeared dead both in furnace and ladle. No aluminum was added. The metal rose in the mold and the ingot was unsound. Much black powder, largely uranium oxide, was taken from the furnace after cooling.

In this heat, part of the ferrouranium was added at the start, plus 18 pounds of crop and skull from heat 1081, the balance at the end. The metal was hot, but poured rather slushy, as if carrying some material in suspension. An oxide skin formed continually in the ladle and while pouring. About 2½ pounds of black powder, mostly uranium oxide, was taken from the furnace after cooling, as well as a few small pea-sized chunks of ferrouranium which had sharp corners, had not been fused, and were identified by their pyrophoric behavior.

TABLE 3.—*Production of uranium*

| No. of heat. | Total weight of charge. | FeU added, analysis. | | | | | Total kw.h. used. | 1st or 2d heat. | FeU added at— | | Size FeU. | Per cent of U. | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Lbs. | No. of sample. | U. | C. | Si. | | | Kw. h. | Start or end. | | Calculated. | Analysis. | |
| | | | | | | | | | | | | | Top. | Butt. |
| | *Lbs.* | | | *P. ct.* | *P. ct.* | *P. ct.* | | | | | | | | |
| 1085 | 46 | 1.75<br>2.75 | 41<br>724 | 45<br>43 | 4.2<br>2.5 | .4<br>2.5 | 30 | 2d... | 0<br>40 | Start<br>End | Hickory.....<br>Pea.......... | [d]2.15<br>[e]2.6<br>4.75 | 1.4 | 1.75 |
| 1086 | 49 | 3 | 41 | 45 | 4.2 | .4 | 37.5 | ...... | 37.5 | In-ladle at end. | Below pea.... | [d].45<br>[e]2.75<br>3.2 | 1.23 | .91 |
| 1087 | 49.5 | 2 | 91 | 68.5 | 5 | .3 | 40 | 1st.. | 40 | In-ladle at end. | ....do........ | [d].7<br>[e]2.7<br>3.4 | .12 | .7 |
| 1228 | 50 | 1.6 | 69 | 37.5 | 1.75 | 2 | 40 | 1st.. | 40 | End | .....do........ | 1.19 | .54 | .51 |
| 1229 | 50 | 1 | 96 | 63 | 2.15 | .8 | 37.5 | 2d... | 37.5 | End | .....do........ | 1.21 | .38 | .34 |
| 1244 | 52.5 | 1.3 | 92 | 63.5 | 2.15 | .7 | 35 | 2d... | 35 | End | Below 8 mesh. | [d].5<br>[e]1.6<br>2.1 | .36 | .32 |
| 1327 | 70.5 | 2 | 68–69 | 37.5 | 2.8 | 2 | 55 | 1st.. | 55 | End | 8 to 30 meshes, no fines. | 1.05 | .36 | .38 |

[d] Start.
[e] End.

## RECOVERY OF URANIUM.

The only really concordant feature of the runs on uranium steel is that the percentage recovery of uranium is low, especially when attempts are made to add much uranium, say 5 per cent or more.

It is also noteworthy that when such attempts are made there is a marked segregation of uranium between the top and the bottom of the ingot. Metallic uranium has a specific gravity of almost 18.5; that is, it is over twice as heavy a material as iron. This alone need not cause segregation, since tungsten, with an even higher specific gravity, does not give trouble from segregation. Uranium and its ferro-alloys oxidize very readily, and in runs 1078 and 1079, when part of the ferro charged at the start was covered with a thick film of oxide, pieces of ferros fairly low in carbon went through the steel furnace without melting or being dissolved.

In heat 1085 some of a clean high-carbon ferro charged at the start also went through unmelted and undissolved. These pieces may readily have acquired an oxide film coating on being charged into the hot furnace before they were covered by molten steel. Even when the ferro is charged into molten steel it may possibly require a surface coating of oxide before it can be submerged under the steel merely by passing through the hot furnace atmosphere, which is *steels in indirect-arc furnace*—Continued.

| Remarks. |
|---|
| Procedure was similar to that of preceding heat, part of the FeU (and 13.3 pounds of heat 1078 crop) being charged at the start, the rest at the end. Metal was not so hot as preceding but less slushy. In addition to the usual black powder, some unfused chunks of ferrouranium were found in the bottom of the furnace. These, by the characteristic fracture of a high-carbon ferrouranium, were recognized as being sample 41, which was added at the start and hence went through the whole heat unfused. None of the lower carbon ferro, 72A, was detected in the material left in the furnace. |
| Aside from 15 pounds of heat 1085 crop charged at the start, the uranium was added as finely crushed ferro in the following manner: The steel was very hot at the start. Half the steel was poured into the ladle, half the ferro added to the ladle, the rest of the steel poured, the rest of the ferro added, the metal stirred, and the whole poured back into the furnace, to mix by pouring, then at once into the ladle again, then immeditately into the mold, since it had become quite cold. The small residue in the bottom of the ladle burnt in the air, throwing off sparks. No uranium oxide or unfused ferro was detected in the furnace. |
| Nineteen pounds heat 1084 crop was charged at the start. The very hot metal was poured into the ladle all the ferrouranium being thrown in when half the steel had been poured, the rest of the steel poured, the ladle stirred, the metal poured back into the furnace, into the ladle, and then into the mold. The metal was still fluid enough for pouring, though it showed signs of a tendency toward slushiness. Some black powder was found in the furnace after cooling. |
| This steel contained 3 per cent Ni. and 1.2 per cent Si. The metal was very hot, the FeU was added in the furnace after the metal was thoroughly skimmed, one "Little Devil" Thermit being used. The metal was fluid and not at all slushy. |
| This steel was similar to the preceding and the procedure was the same. |
| This steel was similar to the two preceding ones, 5 per cent U was contained in the 49 pounds of crop ends of ingots previously made, which were charged at the start, the ferrouranium being added just before pouring. The metal was hot. The ferro was only slowly taken up, but by long and thorough stirring it was apparently taken into solution. No Thermit can was used. |
| Metal was hot. Ferro was readily taken up. This ingot, analyzing 0.45 per cent C, 2.42 per cent Si, 0.70 per cent Mn, 2.92 per cent Ni, 0.37 per cent U, broke up in rolling. |

oxidizing when the furnace is open for charging. Very fine powder, so charged, could be seen to burn before it could be stirred into the metal, and all such very fine material is quite plainly lost. Rather large pieces of ferro were used in the earlier tests for this reason.

The prevalence of slushy heats in which the steel though at a temperature at which W or Mo steels would be fluid as milk, indicates either the probability of the presence of suspended material not in true solution, or of a very wide range between liquidus and solidus in the presence of U. Of the heats which were plainly not slushy—1080, 1082, 1228, 1229, 1244, 1327—only one, 1082, showed appreciable segregation. These contained not over 0.55 per cent U.

Segregation might at first thought always be expected to give more U in the bottom of the ingot than the top, due to the high specific gravity of U, but this need only occur when the steel is poured so hot that the steel remained fluid in the mold long enough to allow settling of U. If the steel is allowed to stand in the ladle for degasification long enough for settling to take place, there would be more U at the bottom of the ladle than at the top. If the steel is then poured into the mold so cold that it freezes before settling can again take place, the top of the ingot, which receives the metal from the bottom of the ladle, might be higher in U.

Savage[19] shows a radiophotograph of a uranium steel containing 7.74 per cent U which has radioluminous patches scattered through the steel in a decidedly nonuniform manner.

Keeney[20] states that steel makers using ferrouranium containing 15 to 30 per cent uranium could not make the uranium stay in high-speed steel, getting from zero to 50 per cent recovery, and that when commercial uranium (a mixture of metallic U and uranium carbide, $U_2C_3$) was used, no better recoveries were made. Experiments were then run in which from 1.42 to 5.45 per cent U was added in 25-pound heats of steel, using ferrouranium ranging from 20 to 40 per cent U, which showed from 9 to 118 per cent recovery of U. He summarizes his results as follows:

*Recovery of uranium.*

| | Per cent recovery. |
|---|---|
| Average of all tests | 54.7 |
| Steel with less than 2 per cent U | 67.8 |
| FeU added after skimming slag | 70.7 |
| FeU added before skimming slag | 48.3 |
| Poured at once after adding FeU | 60.2 |
| Poured one-quarter minute after adding FeU | 45.3 |
| Poured one-half to one minute after adding FeU | 56.7 |

There was no greater variation in the recovery between the use of 20 and 40 per cent U alloys than between two different lots of 25 per cent alloy.

The 9 per cent recovery was obtained when 6.2 pounds of ferro of 20 per cent U was added to about 20 pounds of steel.

In all these runs the ferros were added, in pea or one-half-inch size, either in the furnace just before pouring or in the ladle.

Six tests on 100 to 110 pound heats of crucible tool steel are cited, in which FeU of 50 to 53 per cent U content and 4.7 to 5 per cent C content was used, which show from 16 to 55 per cent recovery of U, averaging 40 per cent.

Nothing is said as to segregation of U, but two of Keeney's tests show, respectively, 100 and 118 per cent recovery by analysis, which makes it appear at least possible that the steels may not have been so sampled that the samples were truly representative if any segregation did occur. If there was segregation the calculated recoveries may not be accurate.

Matthews[21] states that while uranium has been advocated for use in high-speed tool steel, it is difficult to handle, owing to the ease with which it is oxidized, and that steel to which it is added has been more apt to show seams and defects than steel from which it is absent. Microscopic examination, he states, shows the presence of considerable amounts of a material which is probably uranium oxide, since, on account of its specific gravity, the elimination of such oxide is improbable. He says that further experience is needed to allow the introduction of uranium into steel without great loss of the expensive material and without the formation of detrimental impurities. A footnote states that a low-carbon high-uranium ferro recently put on the market should be more successful than the former high-carbon ferro.

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[19] Savage, W., Radioactive luminous materials: Chem. and Met. Eng., vol. 19, 1918, p. 515.

[20] Keeney, R. M., The manufacture of ferro-alloys in the electric furnace: Bull. Am. Inst. Min. Eng., August, 1918, p. 1358 and following.

[21] Matthews, J. A., Modern high-speed steel: Proc. Am. Soc. Test. Mat., vol. 19, pt. 2, 1919, p. 13.

### TESTS OF SILICON-NICKEL-URANIUM STEELS.

Several Si-Ni-U steels were made in 1919 and analyzed as follows:

*Analyses of silicon-nickel-uranium steels.*

| No. of heat. | C. | Si. | Mn. | Ni. | U. |
|---|---|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* |
| 1228 | 0.63 | 1.20 | 0.84 | 3.00 | 0.52 |
| 1229 | .45 | 1.05 | .75 | 3.00 | .36 |
| 1244 | .43 | 1.30 | .90 | 3.00 | 34 |

Foote[22] has since recommended such steels as giving a combination of great static and dynamic strength, hardness, and ductility, and as being suitable for auto gears, light armor, and rivet sets.

Poluskin[23] states that on heats of 400 pounds in an electric furnace, when ferrouranium was added in the furnace at the end of the heat or in the ladle (the latter being preferable), he sometimes got 70 to 75 per cent recovery of U, but that in general the recovery is irregular and under 50 per cent. He states that U oxidizes too readily to allow the addition of ferrouranium to the charge at the beginning of the heat. Poluskin dealt with steels up to about 4 per cent U and with one of 7 per cent. He does not state whether segregation of U was found or if it was looked for, but says that the chemical analyses often had to be repeated several times and the average taken. He finds that there is no advantage in having over 0.5 to 0.6 per cent U in steel, and that any advantage in using U is confined to steels of less than 0.6 per cent C. In a plain U steel of 0.25 to 0.45 per cent C he concludes that U increases the tensile strength and elastic limit without decreasing the ductility, but that it does nothing that can not be accomplished with other alloying elements. He finds no advantage in adding U to Ni or Cr steels. He finds U of no advantage as regards shock tests or repeated impact tests. He states that much of the U in a steel is present as oxide.

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[22] Foote, H. S., U. S. Pat. 1366254: Jan. 18, 1921 (application Sept. 29. 1920).

[23] Poluskin, E., Les aciers á l' uranium: Rev. de Met., vol. 17, 1920, p. 421. Abstracted in Iron Trade Rev., vol. 68, 1921, p. 413, and Iron Age, vol. 106, 1920, p. 1512.

Foote[24] agrees with Poluskin that over 0.6 per cent U gives no advantage, but is more optimistic over the value of small amounts.

The main object in making up the uranium steels listed in Table 3 was to get, if possible, a few steels with at least more than 1 per cent U and as much higher as possible, in order to be able to ascertain what properties fairly large amounts of U confer on steel. Some of the steels should serve for this purpose, although most of them show too much segregation of U to give anything better than a general indication of the properties.

### METHODS OF ADDING URANIUM TO STEEL.

Only tentative conclusions can be drawn as to the most desirable methods of adding U to steel in order to get a good recovery of U and to avoid segregation of U in the steel. It seems probable, however, that at least in making up steels to contain up to about 0.5 per cent U the best results will be obtained by using ferrouranium containing 40 to 65 per cent U with carbon as low as possible, certainly not over 2.5 per cent. It is reasonable to suppose that the presence of, say, 15 per cent silicon in the FeU would be of value in reducing the carbon content of the ferro, though if one assumes a recovery of 40 per cent of the U and 100 per cent of the Si in a ferrouranium of 60 per cent U, 15 per cent Si, 1.5 per cent C, and 22.5 per cent Fe, the addition necessary to give 0.5 per cent U in the steel would raise the silicon by about 0.3 per cent, so that such an alloy could only be used in such types of steel as admit of the presence of a considerable amount of Si. It seems likely, since uranium, like vanadium, forms a carbide with great ease, that further development of ferrouranium and of uranium steels may show that a low-carbon ferro is necessary in order to get the full benefit of the alloying element (if any benefit is conferred by it) as is claimed to be the case with vanadium.[25]

The ferro should probably be crushed to about pea size and the very fine dust screened out, since this is likely to oxidize so readily that it will reach the steel as oxide rather than as metal. On account of the ready oxidation, preheating the ferro appears inadvisable.

In order to get the greatest recovery of U from the ferro, it should be added in the furnace just before pouring or to the stream of metal while pouring, and the steel should be free from slag. While the steel should be given a little time to allow dissemination of the uranium, the shortest time which is compatible with avoidance of segregation between adding the ferro and pouring the steel into the molds will doubtless give the highest recovery of U in the steel.

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[24] Foote, H. S., Uranium as structural steel alloy: Railway Mech. Eng., vol. 94, 1920, p. 695.

[25] Keeney, R. M., The manufacture of ferro-alloys in the electric furnace: Bull. Am. Inst. Min. Eng., 1918, p. 1342.

## ZIRCONIUM STEELS.

The zirconium series of alloy steels was the longest made in the experimental indirect-arc furnace. The use of zirconium steel was brought to the attention of the Navy by W. H. Smith, of the Ford Motor Co.

The Krupp works at Essen were reported[26] to be investigating zirconium steel before the war, and it is known that in 1913, just prior to the war, over 1,000 tons of zirconium ore were shipped from Brazil to Germany, while only about 40 tons were shipped in 1912. Since zirconium oxide began to show its potential value as a refractory about this time, such statistics do not necessarily prove that the ore was intended for the production of zirconium steel. There was also a report[27] in a Brazilian newspaper that the long-range gun that bombarded Paris had a zirconium steel liner.

Ludwig Weiss, of Barmen, Germany, in German Patent 231002, English 29376 of 1910, and U. S. Patent 982326 of January 24, 1911, claims that the disadvantages of titanium in steel—that it is not a sufficiently energetic deoxidizer, that its specific gravity is too low, and that Ti alloys with more than 2 to 3 per cent Ti are difficult to work in the hot state—can be avoided by using zirconium instead of titanium. He claims that zirconium carbide silicide, ferrozirconium, or cuprozirconium can be used for adding Zr to iron, gun metal, brass, copper, aluminum bronze, and the like, and are totally dissolved in them. He advocates alloys of 10 to 25 per cent Zr, and the use of these alloys in such amounts as will add less than 1 per cent Zr (but the patent claims cover adding Zr in minimum amount of 1 per cent, if sound castings are desired, such additions greatly increasing the strength and tenacity of the metal and greater additions further increasing the strength and toughness.

Garcon[28] cites experiments made by the Ford Motor Co. indicating that a steel of 0.42 per cent C, 1 per cent Mn, 1.5 per cent Si, 3 per cent Ni, 0.34 per cent Zr, annealed at 875° C. and air-cooled, reheated to 840° C. and oil-quenched, and drawn in oil at 180° C. for three hours, had a tensile strength of 281,000 pounds, an elastic limit of 240,000 pounds, and a Brinell hardness of 470. The optimum zirconium content is given as one-third of 1 per cent Zr, and it is stated that at above one-half of 1 per cent Zr the quality of the steel is poorer.

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[26] Meyer, H. C., Zirconia, its occurrence and application: Mineral Footnotes, March–April, 1919, p. 11.

[27] Metal Industry (London), Notes of the week: Vol. 13, 1918, p. 91.

[28] Garcon, J., Aciers au zirconium: Bull. Soc. encour. ind. nat., vol. 13, 1919, p. 148. See also Iron Age, Zirconium in steel: Vol. 103, 1919, p. 1015; Foundry, vol. 48, 1920, p. 76.

Tone [29] says, "We hear whispers about a zirconium steel of 300,000 pounds tensile strength and 30 per cent elongation."

It is pointed out [30] that while the combination of high strength, high elastic ratio, high Brinell hardness, and high ductility reported for zirconium steel is unusual, a nickel steel with high silicon and high manganese possesses remarkable quantities in itself, and the action of the zirconium may be merely as an intensifying agent, increasing toughness somewhat as vanadium acts as an intensifying agent on some steels in developing certain properties.

Ramsen [31] states that the value of zirconium steel is probably chiefly as a scavenger. Lantsberry states that it produces a cutting steel of great hardness, but not one possessing any outstanding properties for cutting tools.

Wood [32] states that NiSiZr steel is strongly recommended for parts where extreme hardness and toughness are essential and that it has been used with excellent results for balls and races for large thrust bearings, special heavy duty punches, and rivet sets. He states that the possibilities of this alloy are great.

It is stated [33] that 0.02 per cent Zr may be used as a scavenger in steel, and that zirconium steels have been used in Germany for various purposes.

## COOPERATIVE PRODUCTION OF ZIRCONIUM STEELS.

At the request of the Navy, cooperative work on zirconium steel was undertaken in which the Bureau of Mines was to produce a series of zirconium and other similar steels, which were to be rolled, heat-treated, and subjected to physical tests by the Bureau of Standards. Production of these began in September, 1918, and about 165 steels were made for the Navy. A second series of about 35 steels was later requested and prepared.

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[29] Tone, F. J., Electrochemistry in its human relations, Presidential address: Trans. Am. Electrochem. Soc., vol. 35, 1919, p. 265.

[30] Iron Age: Vol. 103, 1919, p. 1030. Compare also Johnson, C. M., U. S. Pat. 1342911, for a chrome-vanadium-silico-nickel steel.

[31] Ramsen, —, Discussion of paper by Lantsberry, F. C. A. H., Zirconia in relation to the metal industry: Met. Ind., London, vol. 16, 1920, p. 3.

[32] Wood, H. F., Progress in metallurgy of alloy steel: Amer. Drop Forger, Jan., 1920, p. 25.

[33] Jour. du four electrique, Ferro-zirconium: Vol. 29, 1920, p. 157.

According to reports from the Ford Motor Co. it appears that, although zirconium may have a beneficial effect on some steels, it has so far been unable to control the introduction of Zr so as to make steel consistently with the properties which are ascribed to zirconium. That is, three steels may be similarly made and handled and may analyze exactly the same, yet one may not show any changes in physical properties from those given by a similar steel without zirconium, another may show decidedly poorer properties, while a third may have some of its physical properties materially enhanced. It is asserted that the properties can be predicted, before test, from the microstructure, which is claimed to be characteristic when the zirconium has entered the steel in the manner that will give superior physical properties, but that no such prediction can be made without microscopic examination; that is, if zirconium is responsible for the improvement in properties claimed, it gives this improvement only in so-called " freak " heats, which as yet can not be reproduced at will.

At the beginning of this work there seemed reason to hope that zirconium, as well as uranium, might repeat the history of vanadium, which, when first tried out in steel was thought to be useless and condemned by some early investigators; indeed, its value was not apparent until considerable time had elapsed and many trials had been made, through which the correct composition of ferrovanadium, the ways of adding it, the amounts to use, and the other alloying elements in connection with which it is of greatest value were finally found out.

Since the recovery of Zr from ferrozirconium to steel is an important factor in the development of zirconium steel, the experience of the Bureau of Mines in handling various ferros is given below in some detail. (See Table 4.) The only ferros available at first were some made for the Navy by the Thermit reaction (reduction with aluminum) by the Metal and Thermit Co., which gave poor recoveries of Zr.

TABLE 4.—*Recovery of zirconium*

USING THERMIT FeZr OF ABOUT 21 PER CENT Zr, 0.8 PER

| No. of heat. | Total weight charge pounds. | Weight FeZr. pounds. | Total kw.h. used. | 1st or 2d heat. | FeZr added— | Size FeZr. | No. Thermit heating cans used. | Per cent Zr— Calculated. | Per cent Zr— By analysis. Top. | Per cent Zr— By analysis. Butt. | Per cent Zr— By analysis. Average. | Per cent Zr— Recovery. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1134 | 46.5 | 2.2 | 37½ | 2d.. | After 30 kw.h. | Pea.......... | 0 | 0.95 | 0.12 | 0.15 | 0.14 | 0.13 |
| 1145 | 46.5 | 1.4 | 35 | 2d.. | At end....... | .....do....... | 0 | .61 | .14 | .13 | .14 | .23 |
| 1222 | 47.5 | 2.2 | 39 | 1st.. | .....do....... | Under 8 mesh | 2 | .95 | .11 | .07 | .07 | .08 |
| 1223 | 47.5 | 2.2 | 39 | 1st.. | .....do....... | .....do....... | 2 | .95 | .07 | .07 | .07 | .08 |
| Av... | | | | | | | | | | | | .13 |

USING THERMIT FeZr OF ABOUT 16.1 PER CENT Zr, 0.7 PER

| No. of heat. | Total weight charge pounds. | Weight FeZr. pounds. | Total kw.h. used. | 1st or 2d heat. | FeZr added— | Size FeZr. | No. Thermit heating cans used. | Per cent Zr— Calculated. | Per cent Zr— By analysis. Top. | Per cent Zr— By analysis. Butt. | Per cent Zr— By analysis. Average. | Per cent Zr— Recovery. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1101 | 50 | 6 | 40 | 1st.. | At start...... | Hickory nut . | 0 | 2 | 0.09 | 0.1 | 0.1 | 5 |
| 1103 | 50 | 3 | 40 | 1st.. | .....do....... | .....do....... | 0 | 1 | .02 | .02 | .02 | 2 |
| 1105 | 45 | 5.4 | 37 | 1st.. | .....do....... | Pea [a] ........ | 0 | 2 | .07 | .09 | .08 | 4 |
| 1109 | 50 | 6 | 45 | 1st.. | After 40 kw. h. | Pea.......... | 0 | 2 | .07 | .13 | .1 | 5 |
| 1117 | 50 | 2 | 45 | 1st.. | After 37½ kw.h. | .....do....... | 0 | .7 | .09 | .06 | .07 | 10 |
| 1119 | 50 | 3 | 35 | 2d.. | At start...... | Hickory nut to pea. | 0 | 1 | .04 | .07 | .06 | 6 |
| Av... | | | | | | | | | | | | 5 |

USING THERMIT FeZr OF ABOUT 25 PER CENT

| No. of heat. | Total weight charge pounds. | Weight FeZr. pounds. | Total kw.h. used. | 1st or 2d heat. | FeZr added— | Size FeZr. | No. Thermit heating cans used. | Per cent Zr— Calculated. | Per cent Zr— By analysis. Top. | Per cent Zr— By analysis. Butt. | Per cent Zr— By analysis. Average. | Per cent Zr— Recovery. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1231 | 50 | 2.2 | 35 | 2d.. | At end....... | Under 8-mesh | 0 | 1.1 | 0.15 | 0.15 | 0.15 | 0.14 |
| 1241 | 50 | 2.2 | 40 | 1st.. | .....do....... | .....do....... | 0 | 1.1 | .1 | .1 | .1 | 0.09 |
| Av... | | | | | | | | | | | | .11 |

USING THERMIT FeZr OF ABOUT 28.5 PER CENT Zr, 0.5 PER

| No. of heat. | Total weight charge pounds. | Weight FeZr. pounds. | Total kw.h. used. | 1st or 2d heat. | FeZr added— | Size FeZr. | No. Thermit heating cans used. | Per cent Zr— Calculated. | Per cent Zr— By analysis. Top. | Per cent Zr— By analysis. Butt. | Per cent Zr— By analysis. Average. | Per cent Zr— Recovery. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1131 | 45.5 | 1.8 | 40 | 1st.. | After 35 kw. h. | Pea.......... | 0 | 1 | 0.12 | 0.11 | 0.12 | 12 |
| 1132 | 45.5 | 1.8 | 37½ | 2d.. | After 32½ kw.h. | .....do....... | 0 | 1 | .22 | .19 | .2 | 20 |
| 1146 | 46.5 | 1 | 37½ | 1st.. | At end....... | .....do....... | 0 | .6 | .05 | .05 | .05 | 12 |
| 1155 | 46.5 | 1.5 | 40 | 1st.. | .....do....... | .....do....... | 1 | .9 | .11 | .08 | .1 | 11 |
| 1157 | 46.5 | 3.6 | 42½ | 1st.. | .....do....... | 4 to 8 mesh [a]. | 2 | 2 | .09 | .1 | .1 | 5 |
| 1160 | 48.5 | 1.8 | 39 | 1st.. | .....do....... | Under 8-mesh | 0 | 1 | .1 | .1 | .1 | .1 |
| 1161 | 48.5 | 1.8 | 39 | 1st.. | .....do....... | .....do....... | 0 | 1 | .06 | .09 | .08 | 8 |
| 1162 | 48 5 | 1.8 | 37½ | 1st.. | .....do....... | .....do....... | 1 | 1 | .14 | .08 | .11 | 11 |
| 1175 | 50 | 3.6 | 42½ | 1st.. | After 35 kw. h. | .....do....... | 0 | 2 | .1 | .11 | .1 | 5 |
| 1176 | 50 | 3.6 | 37 | 1st.. | At end....... | .....do....... | 2 | 2 | .13 | .16 | .14 | 7 |
| 1177 | 50 | 2.2 | 34 | 2d.. | .....do....... | .....do....... | 1 | 1.3 | .13 | .09 | .1 | .8 |
| Av... | | | | | | | | | | | | 10 |

USING ELECTRO-METALLURGICAL CO.'S FeZr OF 36.3 PER CENT Zr, 0.6 PER

| No. of heat. | Total weight charge pounds. | Weight FeZr. pounds. | Total kw.h. used. | 1st or 2d heat. | FeZr added— | Size FeZr. | No. Thermit heating cans used. | Per cent Zr— Calculated. | Per cent Zr— By analysis. Top. | Per cent Zr— By analysis. Butt. | Per cent Zr— By analysis. Average. | Per cent Zr— Recovery. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1232 | 50 | 1.8 | 40 | 1st.. | At end....... | Under 8-mesh | 0 | 1.25 | .11 | .11 | .11 | 9 |
| 1233 | 50 | 1.8 | 40 | 1st.. | .....do....... | .....do....... | 0 | 1.25 | .16 | .17 | .16 | 1 |
| 1240 | 52 | 1.8 | 37½ | 1st.. | .....do....... | .....do....... | 0 | 1.2 | .12 | .14 | .13 | 11 |
| 1243 | 52 | 1.8 | 45 | 1st.. | .....do....... | .....do....... | 1 | 1.2 | .14 | .14 | .14 | 12 |
| Av... | | | | | | | | | | | | 11 |

USING ELECTRO-METALLURGICAL CO.'S NiZr of 54.9 PER CENT Ni. 25.3 PER CENT

| No. of heat. | Total weight charge pounds. | Weight FeZr. pounds. | Total kw.h. used. | 1st or 2d heat. | FeZr added— | Size FeZr. | No. Thermit heating cans used. | Per cent Zr— Calculated. | Per cent Zr— By analysis. Top. | Per cent Zr— By analysis. Butt. | Per cent Zr— By analysis. Average. | Per cent Zr— Recovery. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1249 | 50 | 2.8 | 37½ | 1st.. | At end....... | Under 8-mesh | 0 | 1.4 | 0.56 | 0.64 | 0.6 | 43 |
| 1290 | 50 | 1.6 | 40 | 1st.. | .....do....... | 4 to 8 mesh... | 0 | .8 | .39 | .4 | .4 | 50 |
| 1291 | 50 | 1.4 | 37½ | 1st.. | .....do....... | Under 8-mesh | 1 | .7 | .23 | .23 | .23 | 33 |
| 1248 | 50 | { [a]2+ .7 } | 43 | 1st.. | .....do....... | .....do....... | 0 | 1.3 | .54 | .61 | .57 | 43 |
| Av... | | | | | | | | | | | | 41 |

*from ferrozirconium to steel.*

**CENT Ti, 10 PER CENT Si, 6 PER CENT Al, 0.2 PER CENT C.**

Notes.

Furnace was opened after metal melted. FeZr was charged and stirred in, then furnace was closed and run 7½ kw. h. more.

Metal was hot enough but FeZr did not go in readily on stirring.

**CENT Ti, 12 PER CENT Si, 3.4 PER CENT Al, 0.7 PER CENT C.**

FeZr was heated just to redness before adding, furnace was closed, and 5 kw. h. more run after adding FeZr. Final steel was rather cool.

FeZr was added cold. Final steel was hot.

Final steel was very hot.

**Zr, Ti n. d. 10 PER CENT, Si, 9 PER CENT Al C. n. d.**

**CENT Ti, 8 PER CENT Si, 3 PER CENT Al, 0.2 PER CENT C.**

Steel was hot, but FeZr did not dissolve readily when stirred.

FeZr was preheated to dull redness; steel was very hot. FeZr did not dissolve readily on stirring.

FeZr was added cold, steel hot.

Some dross was found at end, apparently from FeZr.

Final steel was rather cool.

Final steel was hot enough.

**CENT Ti, 7.5 PER CENT Si, 14.6 PER CENT Al, 1.6 PER CENT Cr, 0.1 PER CENT C.**

**Zr, 0.7 PER CENT Ti, 5.2 PER CENT Si, 7.3 PER CENT Al, 0.1 PER CENT Cr, 0.07 PER CENT C.**

All the Ni was added as NiZr. The steel was very hot and the NiZr well stirred in, but not all was taken up, since the calculated per cent Ni was 3, and that found 1.73 per cent top, 1.73 per cent butt; that is, 58 per cent recovery of Ni.

1.5 per cent Ni was added at start, 1.7 per cent Ni added at end as NiZr; total, 3.2 per cent. Found, 2.97 per cent; that is, 0.23 per cent loss, or a recovery of Ni from that added at the end of 86 per cent.

1.7 per cent Ni was added at start, 1.5 per cent was added at end as NiZr; total, 3.2 per cent. Found, 2.92 per cent; that is, 0.28 per cent loss, or a recovery of Ni from that added at end, of 81 per cent.

Besides NiZr, 0.7 pound of SiZr in heat 1250 was also charged. Metal was very hot. NiZr seemed to go in fairly well. All the Ni charged was carried by the NiZr added at the end, 2.2 per cent Ni being thus added. There was found by analysis 1.62 per cent Ni top, and 1.61 per cent Ni butt, or a recovery of 73 per cent of the Ni.

TABLE 4.—*Recovery of zirconium*

| No. of heat. | Total weight charge pounds. | Weight FeZr. pounds. | Total kw.h. used. | 1st or 2d heat. | FeZr added— | Size FeZr. | No. Thermit heating cans used. | Per cent Zr— | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | Calculated. | By analysis. | | | Recovery. |
| | | | | | | | | | Top. | Butt. | Average. | |
| USING SOUTHERN MANGANESE CO.'S FeZr OF 44.5 PER CENT Zr, 3.2 PER | | | | | | | | | | | | |
| 1292 | 50 | 1.1 | 40 | 1st.. | At end....... | All below 4-mesh, mostly below 8-mesh. | 0 | 1 | 0.05 | 0.05 | 0.05 | 5 |
| 1293 | 50 | 1.1 | 40 | 1st.. | .....do....... | .....do....... | 2 | 1 | .02 | .08 | .05 | 5 |
| Av... | ........ | ........ | ...... | .... | .............. | .............. | ..... | ..... | ...... | ...... | ...... | 5 |
| USING FeZr MADE BY BUREAU OF MINES, OF ABOUT 53 PER | | | | | | | | | | | | |
| 1108 | 50 | 1 | 42 | 1st.. | At end....... | Pea.......... | 0 | 1 | 0.03 | ...... | 0.03 | 3 |
| USING ALLOY MADE BY BUREAU OF MINES, OF ABOUT 64 PER CENT Zr, | | | | | | | | | | | | |
| 1138 | 46.5 | 0.9 | 40 | 1st.. | After 35 kw. h. | Pea.......... | 0 | 1.3 | 0.04 | 0.05 | 0.05 | 4 |
| USING SiZr MADE BY BUREAU OF MINES, OF ABOUT 24 PER CENT Zr, 0. | | | | | | | | | | | | |
| 1133 | 46.5 | 1.8 | 40 | 1st.. | After 35 kw. h. | Pea.......... | 0 | 0.9 | 0.7 | 0.3 | 0.5 | 56 |
| 1134 | 46.5 | 1.1 | 37½ | 1st.. | At end....... | .....do....... | 0 | .6 | .13 | .16 | .15 | 58 |
| USING SiZr MADE BY BUREAU OF MINES, OF ABOUT 36.6 PER | | | | | | | | | | | | |
| 1247 | 52 | 1.8 | 42 | 1st.. | At end....... | Under 8-mesh | 0 | 1.3 | 0.86 | 0.71 | 0.78 | 60 |
| USING SiZr. MADE BY BUREAU OF MINES OF ABOUT 18.9 PER | | | | | | | | | | | | |
| 1250 | 50 | 2.2 | 38 | 1st.. | At end....... | Under 8-mesh | 0 | 0.8 | 0.65 | 0.35 | 0.50 | 63 |
| USING SiZr MADE BY BUREAU OF MINES OF ABOUT | | | | | | | | | | | | |
| 1285 | 50 | 1.6 | 42 | 1st.. | At end....... | Under 8-mesh | 0 | 0.9 | 0.71 | 0.76 | 0.73 | 81 |
| 1289 | 50 | [a]1.6 | 40 | 1st.. | .....do....... | .....do....... | 0 | .7 | .46 | .35 | .40 | 57 |
| Av... | ........ | ........ | ...... | .... | .............. | .............. | 0 | ..... | ...... | ...... | ...... | 69 |

*from ferrozirconium to steel*—Continued.

CENT Ti, 8.7 PER CENT Si, 0.5 PER CENT Al, 3 PER CENT C, 0.36 PER CENT P.

| Notes. |
|---|
| Metal was very hot, but FeZr did not seem to go in very well on stirring. |
| Metal was very hot, but FeZr did not seem to go in very well on stirring, and seemed to be mostly dissolved after Thermit heating cans were used. |

CENT Zr, 0.7 PER CENT Ti, 6 PER CENT Si, 7.7 PER CENT C.

| |
|---|
| Metal was hot, but became rather slushy after FeZr was stirred in. FeZr was not readily dissolved About 0.05 per cent Al was present, so in the crop ends charged at the start no aluminum was added in the ladle. As the metal was slushy it was poured at once without holding it in the ladle to degasify. Metal rose in mold and ingot was unsound. |

1 PER CENT Ti, 3.6 PER CENT Si, 8.6 PER CENT Ni, 6.4 PER CENT C.

| |
|---|
| Furnace was opened and alloy added and stirred in, but alloy was not readily dissolved; furnace was closed and ran 5 kw. h. more. Final metal was very hot, so was held longer than usual in ladle. |

PER CENT Ti 40 PER CENT Si, 0.4 PER CENT Al, 0.1 PER CENT C.

| |
|---|
| Same procedure was followed as on heat 1138, but SiZr was very readily dissolved. |
| SiZr was very readily dissolved. |

CENT Zr, 1.3 PER CENT Ti, 44.5 PER CENT Si, 0.7 PER CENT Al.

CENT Zr, 0.8 PER CENT Ti, 34.2 PER CENT Si, 1.6 PER CENT Al.

20 PER CENT Zr, 0.3 PER CENT Ti, 40 PER CENT Si.

| |
|---|
| SiZr was made up mainly of that of heats 1285 and 1289, with small amounts of SiZr alloys of heats 1247 and 1250. |

TABLE 4.—*Recovery of zirconium*

USING ELECTRO-METALLURGICAL CO.'S SiZr OF 29.8 PER

| No. of heat. | Total weight charge pounds. | Weight FeZr. pounds. | Total kw.h. used. | 1st or 2d heat. | FeZr added— | Size FeZr. | No. Thermit heating cans used. | Per cent Zr— |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  |  |  |  |  | Calculated. | By analysis. |  |  | Recovery. |
|  |  |  |  |  |  |  |  |  | Top. | Butt. | Average. |  |
| 1180 | 48.5 | 1.6 | 37 | 1st.. | At end....... | Under 8-mesh | 0 | 1 | 0.48 | 0.48 | 0.48 | 48 |
| 1181 | 48.5 | 1.6 | 35 | 1st.. | .....do....... | 4 to 8 mesh... | 0 | 1 | .61 | .6 | .6 | 60 |
| 1182 | 48.5 | 1.6 | 32½ | 2d.. | At start...... | Egg.......... | 0 | 1 | None. | .03 | .02 | 2 |
| 1183 | 48.5 | 1.6 | 37½ | 1st.. | After32½kw.h. | .....do....... | 0 | 1 | .23 | .22 | .23 | 23 |
| 1184 | 48.5 | 1.1 | 34 | 2d.. | At end....... | Under 8-mesh | 0 | .7 | .29 | .24 | .27 | 47 |
| 1185 | 48.5 | 1.1 | 35 | 1st.. | .....do....... | .....do....... | 0 | .7 | .5 | .3 | .4 | 57 |
| 1186 | 48.5 | 1.1 | 34 | 2d.. | .....do....... | .....do....... | 0 | .7 | .24 | .26 | .25 | 36 |
| 1187 | 48.5 | 1.1 | 35 | 1st.. | .....do....... | .....do....... | 0 | .7 | .38 | .27 | .33 | 33 |
| 1188 | 48.5 | 1.1 | 35 | 1st.. | .....do....... | .....do....... | 0 | .7 | .33 | .31 | .32 | 46 |
| 1189 | 48.5 | 1.1 | 35 | 1st.. | .....do....... | .....do....... | 0 | .7 | .15 | .25 | .2 | 29 |
| 1190 | 48.5 | 1.4 | 33 | 2d.. | .....do....... | .....do....... | 0 | .9 | .47 | .44 | .46 | 51 |
| 1191 | 48.5 | 1.4 | 35 | 1st.. | .....do....... | .....do....... | 0 | .9 | .4 | .38 | .39 | 43 |
| 1192 | 48.5 | [a]1.4 | 33 | 2d.. | .....do....... | .....do....... | 0 | .9 | .8 | .35 | .58 | 65 |
| 1193 | 48.5 | [a]1.4 | 35 | 1st.. | .....do....... | .....do....... | 1 | .9 | .3 | .34 | .3 | 33 |
| 1194 | 48.5 | 1.4 | 33 | 2d.. | .....do....... | .....do....... | 0 | .9 | .45 | .45 | .45 | 50 |
| 1195 | 48.5 | 1.4 | 35 | 1st.. | .....do....... | .....do....... | 0 | .9 | .51 | .5 | .5 | 56 |
| 1196 | 48.5 | 1.4 | 35 | 1st.. | .....do....... | .....do....... | 0 | .9 | .6 | .48 | .54 | 60 |
| 1197 | 48.5 | [a]1 4 | 33 | 2d.. | .....do....... | .....do....... | 0 | .9 | .32 | .34 | .33 | 38 |
| 1210 | 48.5 | 1.3 | 32½ | 2d.. | .....do....... | Under 4-mesh | 0 | .8 | .55 | .55 | .55 | 69 |
| 1211 | 48.5 | 1.3 | 35 | 1st.. | .....do....... | .....do....... | 0 | .8 | .8 | .3 | .55 | 69 |
| 1212 | 48.5 | .9 | 33 | 2d.. | .....do....... | .....do....... | 0 | .55 | .4 | .2 | .3 | 55 |
| 1213 | 48.5 | .9 | 31 | 1st.. | .....do....... | .....do....... | 0 | .55 | .31 | .33 | .32 | 58 |
| 1218 | 48.5 | [a]1.3 | 40 | 1st.. | .....do....... | Under 8-mesh | 2 | .8 | .8 | .3 | .55 | 69 |
| 1219 | 48.5 | [a]1.3 | 37½ | 1st.. | .....do....... | .....do....... | 1 | .8 | .71 | .72 | .72 | 90 |
| 1220 | 48.5 | [a].65 | 35½ | 2d.. | .....do....... | .....do....... | 1 | .4 | .22 | .18 | .2 | 50 |
| 1221 | 48.5 | [a].65 | 37½ | 2d.. | .....do....... | .....do....... | 2 | .4 | .17 | .17 | .17 | 43 |
| 1224 | 48.5 | [a]1.3 | 37½ | 2d.. | .....do....... | .....do....... | 0 | .8 | .18 | .23 | .2 | 25 |
| 1225 | 48.5 | [a]1.3 | 37½ | 1st.. | .....do....... | .....do....... | 0 | .8 | .18 | .22 | .2 | 25 |
| Av... | ........ | ........ | ........ | ........ | ........ | ........ | ........ | ........ | ........ | ........ | ........ | 56 |

[a] See note.

[b] Average, omitting Nos. 1182 and 1183.

## RECOVERY OF ZIRCONIUM.

Table 4 (see p. 44) indicates that alloys ranging from 16 to 36 per cent Zr, 0.5 to 0.7 per cent Ti, 7.5 to 12 per cent Si, 3.5 to 14.5 per cent Al, and 0.1 to 0.7 per cent C, made from the ore by reduction with aluminum by the Thermit reaction, not in an electric furnace, all gave low recoveries of Zr, averaging only 10 per cent, no matter how they were added. These ferros would lie on top of a cleanly skimmed bath of very hot steel without going into solution readily, even when vigorously stirred, and would bob back to the surface, doubtless becoming oxidized while remaining on the surface, since the alloys took on a surface film of oxide when attempts were made to preheat them, as in run 1157 (see note, p. 45, Table 4).

Table 4 (see p. 44) shows that the recovery of Zr when a FeZr high in carbon is used was still lower, under 5 per cent. This might be expected from the analogy with ferro-carbon-titanium (16 per cent Ti, 7.5 per cent C) with which it is said to be almost impossible to make more than traces of Ti remain in the steel, the alloy being used only as a scavenger.

*from ferozirconium to steel*—Continued.

CENT Zr, 3.1 PER CENT Ti, 46.7 PER CENT Si ,0.03 PER CENT C.

| Notes. |
|---|
| Surface of ingot was rather poor. |
| |
| |
| |
| |
| |
| |
| |
| |
| |
| |
| |
| FeTi as well as SiZr was added to these heats (see Table 5). |
| Do. |
| |
| |
| |
| Do. |
| |
| |
| |
| Metal was rather cold. |
| FeTi as well as SiZr was added to these heats (see Table 5). |
| Do. |
| Do. |
| Do. |
| Do. |
| Do. |

On the other hand, when a FeZr of 13 per cent Zr, 1 per cent Ti, 4 per cent Si, 5 per cent C, 0.9 per cent Al is used for addition to the ladle, the following recoveries were said to have been made by the Ford Motor Co.

*Recoveries of zirconium in three heats.*

| Heat No. | Zr added. | Zr found. | Recovery of Zr. |
|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* |
| 901-2 | 0.25 | 0.20 | 80 |
| 901-3 | .50 | .35 | 70 |
| 901-4 | 1 | .54 | 54 |

Table 4 (see p. 44) shows that a NiZr alloy (made by reduction with Al in an electric furnace), although similar in composition (save that Fe was mostly replaced by Ni) to the FeZr's made by Al reduction, gave a distinctly better recovery of Zr. That only a 60 to 80 per cent recovery of the Ni contained in the alloy was made (see notes 12 to 15) shows that there is a mechanical loss of alloy. This may possibly be due to some of the alloy floating on the steel before it goes into solution, being pushed to the edges of the furnace in stirring, and there sticking to the rim of slag which remains after skimming, and being then either scraped out with the slag or part of it left in the furnace until the next heat. This is quite possible, in heat 1249 at least, as the following figures show:

*Per cent of nickel calculated and found in two consecutive heats.*

| No. of heat. | Per cent Ni— | | Loss. | Gain. |
|---|---|---|---|---|
| | Calculated. | Found. | | |
| 1249..... | 3 | 1.73 | 1.27 | .......... |
| 1250..... | 3 | 3.57 | .......... | 0.57 |

Mechanical loss may also occur through the presence of extremely fine powder, which if not sifted out after the ferro is crushed down to the desired size would float more readily than the larger particles and would thus be on the surface longer before being stirred in, and would have more surface area in comparison with its weight, so that oxidation of the surface is quite probable. If the surface does become oxidized, such small particles would be relatively slow to coalesce with the main mass of metal, even if they should melt; if they should not melt there may not be enough agitation in stirring to break through the oxide film and expose a clean metal surface to the dissolving action of the bath of steel.

Table 4 shows that silicozirconium alloys were readily taken into the bath and gave an average recovery of 50 to 60 per cent. These alloys go into the steel as readily, as far as the eye can tell, as the same amount of ferrosilicon. Since silicon is so very readily soluble in a hot bath of steel, the steel may have first dissolved the silicon, thus disintegrating the alloy and wetting it at the same time, so that the suspension of clean particles of alloy higher in Zr and lower in Si may occur as soon as the solution of Si has well started.

The differences in the solubility of the various alloys in steel may be closely related to the melting point. The Bureau of Standards has planned an investigation of the melting points of various ferro-alloys used for making alloy steels, and at the request of that bureau samples of representative alloys used in making the steels cited in this report have been sent to it for study. When the Bureau of Standards issues a report on its melting-point work, it will probably throw a good deal of light on the question of recovery of the various elements from their ferro-alloys into steel.

A better recovery can probably be made in pouring steel in commercial quantities into large ladles by throwing the crushed ferro into the stream of metal as it flows from furnace to ladle, as the pieces would become submerged nad be carried along by the stream. The design of the experimental indirect-arc furnace made it impossible to reach under the furnace and do this.

Although SiZr gives the highest recovery of any of the Zr alloys, when added at the end, it will be noted that on heat 1182, when SiZr to give 1 per cent Zr was added at the start of the heat, none was found in the finished steel. This is checked by heats 1204 and 1205, in which crop ends of Zr steels were remelted, the data being shown below.

*Results of remelting crop ends of zirconium steels.*

| Heat No. | Present in crop ends at start. | | Analysis of finished steel. | | | |
|---|---|---|---|---|---|---|
| | Per cent Zr. | Per cent Ti. | Per cent Zr. | | Per cent Ti. | |
| | | | Top. | Butt. | Top. | Butt. |
| 1204 | 0.25 | 0.03 | None. | None. | 0.005 | 0.005 |
| 1205 | .20 | .03 | None. | None. | .008 | .008 |

All the Zr was removed, but a small amount of Ti remained in the steel, even though the amount of Zr present in the charge was much greater than that of Ti.

The relatively good recovery of Zr from a SiZr alloy found in the series noted above was checked up by the later series on which data are given below. The good recovery from a NiZr alloy made by reduction with aluminum (in the electric furnace) was also checked. It is hard to see why NiZr alloys thus made are readily absorbed and give a good recovery of Zr whereas FeZr alloys thus made give a low recovery, but this difference was shown consistently.

The NiSiZr alloy was used in the hope that the good recovery of the SiZr alloy could be combined with freedom from segregation, since in the first series the NiZr alloy gave steels that were not segregated, but in the second series neither the NiZr nor NiSiZr alloys gave steels in which Zr did not segregate if the Zr content was high.

*Recovery and segregation of zirconium in second series of zirconium steels.*

[Steel, 0.35 to 0.50 per cent C; Ni, 3 per cent; Si, 1.6 to 2.7 per cent; Mn, 0.55 to 0.75 per cent; S, below 0.04 per cent; P, below 0.03 per cent.]

HEAT 1317, REMELT OF ZR STEEL (CROP ENDS).

| | C. | Si. | Mn. | Ni. | Al. | Ti. | Zr. |
|---|---|---|---|---|---|---|---|
| Calculated per cent | 0.47 | 2.76 | 0.74 | 2.93 | 0.03 | 0.034 | 0.44 |
| Found do | .40 | 2.73 | .57 | 2.95 | .01 | .01 | .03 |
| Recovery do | .85 | .99 | .77 | 100 | 30 | 30 | 7 |

### ALLOY USED—SILICOZIRCONIUM.

[Zr, 29.84; Si, 46.68; Fe, 18.75; Ti, 3.07; C, 0.03; P, 0.12; S, 0.03.]

| No. of heat. | Calculated, per cent. | | Found, per cent. | | | | Recovery, per cent. | |
|---|---|---|---|---|---|---|---|---|
| | Ti. | Zr. | Ti. | Zr. | | | Ti. | Zr. |
| | | | | Top. | Butt. | Average. | | |
| 1304 | 0.025 | 0.25 | 0.02 | 0.11 | 0.13 | 0.12 | 80 | 50 |
| 1305 | .04 | .40 | .035 | .19 | .23 | .20 | 86 | 50 |
| 1306 | .06 | .60 | .03 | .26 | .22 | .24 | 50 | 40 |
| 1307 | .08 | .80 | .05 | .55 | .35 | .45 | 62 | 56 |
| 1308 | .10 | 1 | .07 | .70 | .45 | .57 | 70 | 57 |
| 1309 | .05 | .49 | .02 | .26 | .23 | .25 | 40 | 50 |
| 1310 | .075 | .75 | .04 | .55 | .38 | .46 | 53 | 61 |
| 1322 | .025 | .25 | .02 | .12 | .11 | .11 | 80 | 45 |
| 1323 | .08 | .80 | .07 | .50 | .40 | .45 | 87 | 56 |
| 1324 | .10 | 1 | .09 | .65 | .45 | .55 | 90 | 55 |
| Average | | | | | | | 70 | 52 |

### ALLOY USED—NICKEL-ZIRCONIUM.

[Zr, 25.29; Si, 5.20; Fe, 6.35; Ti, 0.65; C, 0.07; Al, 7.34; Ni, 54.90; Cr, 0.10; P, 0.066; S, 0.01.]

| No. of heat. | Calculated, per cent. | | | Found, per cent. | | | | | Recovery, per cent. | | | Ni added as— | | | Ni found. | Ni lost. | Ni as Ni Zr minus Ni loss. | Per cent Ni added as Ni Zr recovered. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Al. | Ti. | Zr. | Al. | Ti. | Zr. | | | Al. | Ti. | Zr. | Ni. | NiZr. | Sum. | | | | |
| | | | | | | Top. | Butt. | Av. | | | | | | | | | | |
| | | | | | | | | | | | | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* |
| 1311 | 0.17 | 0.015 | 0.60 | 0.06 | 0.01 | 0.38 | 0.21 | 0.30 | 35 | 67 | 50 | 1.90 | 1.30 | 3.20 | 2.97 | 0.23 | 1.07 | 82 |
| 1312 | .23 | .03 | .80 | .19 | .02 | .45 | .32 | .38 | 83 | 67 | 48 | 1.52 | 1.73 | 3.25 | 2.97 | .28 | 1.45 | 84 |
| 1313 | .07 | .005 | .25 | .02 | .01 | .11 | .11 | .11 | 29 | 100 | 44 | 2.55 | .55 | 3.10 | 2.89 | .21 | .34 | 62 |
| 1314 | .13 | .015 | .45 | .08 | .015 | .24 | .17 | .20 | 61 | 100 | 45 | 2.17 | .98 | 3.15 | 2.95 | .20 | .78 | 80 |
| 1315 | .28 | .02 | .66 | .12 | .02 | .45 | .26 | .35 | 43 | 100 | 53 | 1.78 | 1.42 | 3.20 | 2.84 | .36 | 1.06 | 75 |
| Average | | | | | | | | | 50 | 86 | 48 | | | | | | | 79 |

### ALLOY USED—NICKEL-SILICON-ZIRCONIUM.

[Zr, 27.17; Si, 35.21; Ni, 22.28; Ti, 1.40; Al, 2.54; Cr, 0.50; C, 0.14; Fe, 10.20.]

| No. of heat. | Calculated, per cent. | | | Found, per cent. | | | | | Recovery, per cent. | | | Ni added as— | | | Ni found. | Ni lost. | Ni as NiSiZr minus Ni loss. | Per cent Ni added as NiSiZr recovered. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Al. | Ti. | Zr. | Al. | Ti. | Zr. | | | Al. | Ti. | Zr. | Ni. | NiSiZr. | Sum. | | | | |
| | | | | | | Top. | Butt. | Av. | | | | | | | | | | |
| | | | | | | | | | | | | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* |
| 1316 | 0.065 | 0.035 | 0.70 | 0.02 | 0.03 | 0.61 | 0.38 | 0.50 | 31 | 86 | 71 | 2.52 | 0.58 | 3.10 | 2.98 | 0.12 | 0.46 | 80 |
| 1318 | .05 | .025 | .50 | .02 | .02 | .40 | .25 | .32 | 40 | 80 | 64 | 2.64 | .42 | 3.06 | 2.94 | .10 | .32 | 76 |
| 1319 | .065 | .035 | .70 | .01 | .03 | .55 | .33 | .44 | 16 | 86 | 63 | 2.52 | .58 | 3.10 | 3.07 | .03 | .55 | 95 |
| 1320 | .085 | .045 | .90 | .02 | .04 | .65 | .50 | .57 | 24 | 89 | 63 | 2.44 | .61 | 3.15 | 3.05 | .10 | .51 | 84 |
| 1321 | .10 | .06 | 1.10 | .04 | .05 | .90 | .60 | .75 | 40 | 83 | 68 | 2.30 | .90 | 3.20 | 2.96 | .24 | .66 | 74 |
| Average | | | | | | | | | 30 | 85 | 66 | | | | | | | 80 |

### AVERAGE RECOVERY OF ALL ALLOYS.

| | Per cent. |
|---|---|
| Ti | 79 |
| Zr | 52 |
| Ni | 80 |
| Al | 40 |

The crushed alloys were added in the electric furnace at the end of the heat after the slag was carefuly skimmed, were immediately stirred in, and the steel then poured into the ladle.

Since Ni is quantitatively recovered, once it gets into the bath, and since there is but an 80 per cent recovery of Ni from the Zr alloy, it appears that about 20 per cent of the alloy added is mechanically lost, either by oxidation of the finer particles (the alloys were crushed to pea size and below and carried considerable fines) before it hits the steel so that the oxidized particles are not dissolved, or else is caught in traces of slag not skimmed off.

Ti averages an 80 per cent recovery also; that is, it appears that all the Ti in the alloy that hits the steel is recovered.

The SiZr and the NiZr alloys show a Zr recovery of 50 per cent; that is, about 62 per cent of the Zr in the alloy hitting the steel is recovered. The percentage loss does not vary directly with the amount added, but is quite constant, whether one-fourth or 1 per cent Zr is added.

The NiSiZr alloy shows a Zr recovery of about 65 per cent, over 80 per cent of the Zr in the alloy hitting the steel being recovered, the percentage loss being practically the same whether one-half or 1 per cent be added.

Al shows a recovery of 50 per cent from an alloy carrying 7.3 per cent Al, and of 30 per cent from one carrying 2.5 per cent Al.

When more than 0.5 per cent Zr is added (probably 0.4 per cent on the NiSiZr) and more than 0.25 per cent Zr recovered, there is a very marked segregation, even in these small 66 to 72 pound 6 by 3 inch ingots. There is no proof that the *average* Zr content, figured from top and butt analyses, is the real average, though it has been used, for want of better data, in the above calculation of recoveries. It is quite possible that the butt figure may come nearer to the actual recovery than the average, since the butt sample was taken from the bottom, which should freeze very quickly, and represent the Zr content of the melt. Segregation could not be studied on the ingot without ruining the ingot for rolling. The segregation is a true segregation, not analytical errors, for duplicate analyses from the same top or butt sample agreed within 0.05 per cent Zr or less.

Recovery figures on Ti and Al show the order of magnitude rather than the exact recovery, due to the large effect on the calculations of a difference of 0.01 per cent in these elements. The analyses usually checked to less than 0.01, but reporting figures to 0.001 or even 0.005 on these elements is hardly justified analytically, and not necessary for utilization of the figures.

Zr segregation appears to be the same with any of the alloys, and seems negligible if the final steel contains not over 0.25 per cent Zr. The remelt of Zr steel (heat 1317) shows, as have previous tests on the former series, that on remelting, even in an electric furnace without running either oxidizing or reducing slags, the Zr is almost entirely lost.

Although better recoveries of Zr from each type of zirconium alloy may be expected in commercial practice, it is probable that the order of recovery from each type of alloy will remain the same in these experiments.

Not only is the recovery of Zr from Zr alloy to steel much better from the SiZr than from the FeZr alloys made by Al reduction but the recovery of Zr from ore to alloy is higher.

In making up a number of FeZr alloys from baddeleyite ore by the thermit reaction, the Metal & Thermit Corp. got recoveries as follows:

*Recovery of zirconium.*

| Composition of alloy per cent. | | | Per cent recovery Zr ore to alloy. |
|---|---|---|---|
| Zr. | Si. | Al. | |
| 16.9 | 12.3 | 3.5 | 25 |
| 16.4 | 10 | 4.2 | 8 |
| 25.6 | 10 | 9.1 | 40 |
| 14.2 | 7.5 | 10 | 11 |

The Electro Metallurgical Co. in making alloys of the type 30 per cent Zr, 15 per cent Si, 10 per cent Al got a recovery from the ore of about 50 per cent. The recovery in making the NiZr alloy would be expected to be about the same. In refining this to reduce the Al, and in the previous treatment of the ore to lower the Si so as to make a final alloy of 30 per cent Zr, 5 per cent Si, and 2 per cent Al, the recovery would fall to 35 per cent.

In making the SiZr alloy on a commercial scale, however, the recovery from the ore is 80 per cent or better. The recovery of Zr from ore to ferro in making a FeZr by carbon reduction is not known, but will probably not be over 80 per cent. From the recovery of Zr from ore to alloy and that from alloy to steel we can calculate the recovery from ore to steel.

*Recovery of zirconium from ore to steel.*

| Alloy. | | | | | Per cent recovery Zr— | | |
|---|---|---|---|---|---|---|---|
| Zr. | Si. | Ni. | Al. | C. | Ore to alloy. | Alloy to steel. | Ore to steel |
| 30 | 15 | .......... | 10 | Tr. | 50 | 15 | 7.5 |
| 25 | 5 | 55 | 7 | Tr. | [a] 50 | 45 | 23 |
| 30 | 5 | .......... | 5 | Tr. | 35 | 10 | 3.6 |
| 30 | 45 | .......... | .......... | Tr. | 80 | 55 | 44 |
| 45 | 8 | .......... | .5 | 3 | [b] 80 | 4 | 3 |

[a] Or less.

[b] Assumed.

The SiZr alloy is decidedly more promising than any of the others from the point of view of recovery, but this, of course, is only admissible for use in steels which may contain a good deal of Si in proportion to the amount of Zr desired.

The segregation of Zr is not accompanied by a similar segregation of Si in steels made by the use of SiZr or SiNiZr.

*Recovery of zirconium and silicon from steels.*

| No. of heat. | Zr. | | | Si. | | |
|---|---|---|---|---|---|---|
| | Top. | Butt. | Difference. | Top. | Butt. | Difference. |
| 1133 | 0.70 | 0.30 | +0.40 | 1.66 | 1.64 | +0.02 |
| 1185 | .50 | .30 | + .20 | 1.41 | 1.36 | + .05 |
| 1187 | .38 | .27 | + .11 | 1.44 | 1.31 | + .13 |
| 1192 | .80 | .35 | + .45 | 1.49 | 1.48 | + .01 |
| 1196 | .60 | .48 | + .12 | 1.50 | 1.49 | + .01 |
| 1211 | .80 | .30 | + .50 | 1.18 | 1.07 | + .11 |
| 1212 | .40 | .20 | + .20 | .73 | .81 | − .08 |
| 1250 | .65 | .35 | + .30 | 1.54 | 1.56 | − .02 |
| 1307 | .55 | .35 | + .20 | 2.50 | 2.42 | + .08 |
| 1308 | .70 | .45 | + .25 | 2.76 | 2.74 | + .02 |
| 1310 | .55 | .38 | + .17 | 1.95 | 1.85 | + .10 |
| 1323 | .50 | .40 | + .10 | 2.46 | 2.31 | + .15 |
| 1324 | .65 | .45 | + .20 | 2.68 | 2.68 | .00 |
| 1316 | .61 | .38 | + .23 | 1.79 | 1.82 | − .03 |
| 1318 | .40 | .25 | + .15 | 2.54 | 2.53 | + .01 |
| 1319 | .55 | .33 | + .22 | 2.25 | 2.02 | + .23 |
| 1320 | .65 | .50 | + .15 | 2.46 | 2.51 | − .05 |
| 1321 | .90 | .60 | + .30 | 2.10 | 1.98 | + .12 |
| Average | | | + .34 | | | + .05 |

There was about one and one-half times as much Si as there was Zr in these alloys used for adding Zr, so that if the segregation of Zr was due to undissolved particles of the alloy, the Si segregation should be greater than one-seventh as much as the segregation of Zr. It is very evident that the Zr alone, out of the SiZr, is segregated. Comparison of the segregated and uniform Zr steels as to C, Si, Mn, and Ni content shows that these elements vary as much or more in uniform Zr as in segregated Zr steels.

The only uniformity shown by the segregated Zr steels is that when segregation does occur the Zr is higher in the top of the ingot than in the butt.

It should be noted that, like titanium,[34] zirconium, even in annealed steel, interferes with the determination of sulphur by the evolution method, giving low results.

On some steels in Table 5 (see p. 56) Ti was added as ferrotitanium in addition to the small amounts present in the zirconium alloys used. The ferrotitanium used was made by the Thermit reaction and obtained from the Metal & Thermit Co. It contained about 23.6 per cent Ti, 1.4 per cent Si, and 5.4 per cent Al.

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[34] **Lord, N. W., and Demorest, D. J., Metallurgical analysis: New York, 1916, p. 98.**

### TABLE 5.—*Titanium-zirconium steels.*

| No. of heat. | Total weight charge. | Weight SiZr added. | Weight FeTi added. | Kw. h. used. | 1st or 2d heat. | FeTi and SiZr added— | Number Thermit heating cans used. | Size FeTi. | Per cent Ti— Calculated.[a] | Per cent Ti— Found. Top. | Per cent Ti— Found. Butt. | Per cent Ti— Found. Av. | Per cent Ti— Recovered. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *Lbs.* | *Lbs.* | *Lbs.* | | | | | | | | | | |
| 1192 | 48.5 | 1.4 | 0.33 | 33 | 2d | At end | 0 | Under 8-mesh | 0.15 | 0.15 | 0.15 | 0.15 | 60 |
| 1193 | 48.5 | 1.4 | .33 | 35 | 1st | do | 1 | do | .25 | .18 | .23 | .2 | 80 |
| 1196 | 48.5 | 1.4 | .33 | 35 | 1st | do | 0 | do | .25 | .12 | .14 | .13 | 50 |
| 1197 | 48.5 | 1.4 | .33 | 33 | 2d | do | 0 | do | .25 | .17 | .15 | .16 | 64 |
| 1218 | 48.5 | 1.3 | .66 | 40 | 1st | do | 2 | do | .4 | .4 | .28 | .36 | 88 |
| 1219 | 48.5 | 1.3 | .66 | 37½ | 1st | do | 1 | do | .4 | .16 | .14 | .15 | 37 |
| 1224 | 48.5 | 1.3 | .33 | 37½ | 2d | do | 0 | do | .24 | .1 | .1 | .1 | 41 |
| 1225 | 48.5 | 1.3 | .33 | 37½ | 1st | do | 0 | do | .24 | .11 | .11 | .11 | 46 |
| Average | | | | | | | | | | | | | 58 |

[a] Includes Ti in SiZr.

### TABLE 6.—*Titanium steels.*

The following data have been obtained on titanium steels, using FeTi of about 23.6 per cent Ti, 1.4 per cent Si, 5.4 per cent Al.]

| No. of heat. | Total weight charge. | Weight FeTi. | Kw. h. used. | 1st or 2d heat. | FeTi added. | Size FeTi. | No. Thermit cans used. | Per cent Ti— Calculated. | Per cent Ti— Found. Top. | Per cent Ti— Found. Butt. | Per cent Ti— Found. Av. | Per cent Ti— Recovered. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *Lbs.* | *Lbs.* | | | | | | | | | | |
| 1098 | 50 | 1.75<br>.45<br>2.2 | 40 | 1st | At start<br>At end | Pea | 0 | .9<br>.2<br>1.1 | 0.29 | 0.22 | 0.25 | 23 |
| 1099 | 50 | 3.95<br>.45<br>4.4 | 40 | 1st | At start<br>At end | do | 0 | 2<br>.2<br>2.2 | .4 | .32 | .36 | 14 |
| 1100 [a] | 50 | 4.4 | 45 | 1st | At end | do | 0 | 2.2 | .97 | .98 | .98 | 44 |
| 1102 | 50 | 0.22 | 37½ | 1st | do | do | 0 | .1 | .06 | .06 | .06 | 60 |
| 1104 | 50 | 0.11 | 37½ | 1st | do | do | 0 | .05 | .04 | .03 | .03 | 60 |
| 1110 | 50 | 5.5 | 45 | 2d | | do | 0 | 2.6 | .54 | .54 | .54 | 21 |
| 1113 [b] | 50 | 0.11 | 37½ | 1st | After 35 kw. h. | do | 0 | .05 | .03 | .04 | .03 | 60 |
| 1116 [c] | 50 | 5.5 | 45 | 1st | do | do | 0 | 2.6 | .54 | .44 | .49 | 19 |
| 1118 | 49.5 | 1.05 | 42½ | 1st | At end | do | 0 | .5 | .32 | .38 | .35 | 70 |
| 1130 | 50 | 0.8 | 37½ | 1st | do | do | 0 | .4 | .32 | .35 | .33 | 82 |
| 1148 [d] | 46.5 | 4.4 | 40 | 1st | do | do | 1 | 2.3 | 1.45 | 1.53 | 1.49 | 65 |
| 1149 | 46.5 | 2.75 | 40 | 1st | do | do | 1 | 1.4 | .89 | .92 | .90 | 64 |
| 1150 | 46.5 | 1.45 | 42½ | 1st | do | do | 1 | .75 | .53 | .15 | .34 | 50 |
| 1151 | 48.5 | 2.75 | 40 | 1st | do | do | 1 | 1.4 | 1.04 | n. d. | | 74 |
| 1152 [e] | 46.5 | 2.75 | 40 | 1st | do | do | 2 | 1.4 | .66 | 1.05 | .85 | 60 |
| 1153 [f] | 46.5 | 4.9 | 42½ | 1st | do | do | 2 | 2.5 | 1.7 | 2.03 | 1.86 | 74 |
| 1174 | 48.5 | 1.25 | 35 | 2d | do | Under 8-mesh | 0 | .6 | .28 | .26 | .27 | 45 |
| 1179 [g] | 46.5 | 2.75 | 40 | 1st | do | do | 2 | 1.4 | .76 | .71 | .73 | 52 |
| 1216 | 48.5 | 1.65 | 33 | 1st | do | do | 1 | .8 | .45 | .44 | .45 | 56 |
| 1217 [h] | 48.5 | 1.65 | 35 | 1st | do | do | 2 | .8 | .49 | .40 | .45 | 56 |
| 1242 [i] | 50 | 2.6 | 37½ | 1st | do | do | 1 | 1.2 | .59 | .62 | .60 | 50 |
| 1246 | 50 | 2.2 | 42½ | 1st | do | do | 2 | 1 | .45 | .44 | .44 | 44 |
| 1269 | 50 | 2.2 | 35 | 2d | do | do | 1 | 1 | .67 | .62 | .65 | 69 |
| 1284 | 50 | 1.65 | 37½ | 2d | do | do | 2 | .8 | .56 | .54 | .65 | 69 |
| Average | | | | | | | | | | | | 65 |

[a] Metal was only normally hot. FeTi did not dissolve readily, but lumped together into a ball. It was stirred around as long as possible, but it was still undissolved when the steel was so cold it had to be poured into the ladle and then at once to the mold. Two and three-fourths pounds lump of FeTi plus adhering steel remained in furnace after pouring.

[b] FeTi was added after 35 kw. h., then furnace was closed and run 10 kw. h. more.

[c] FeTi preheated before charging, added after 35 kw. h., then 10 kw. h. more run, about 2 pounds FeTi skimmed off undissolved.

[d] Metal was very hot, surface was skimmed perfectly clean and FeTi added and stirred. Charge was rather pasty, so Thermit heating can was used and metal was poured by working fast.

[e] Metal was not very hot. Fluid poured into ladle at start, but was slushy at end, though not appreciably colder.

[f] FeTi was preheated to dull red; metal not very slushy at end.

[g] Steel was faintest trace slushy at very last of pouring into mold.

[h] Owing to lining coming off and covering bath, steel was not very hot.

[i] Same conditions as on heat 1242 existed as to lining and temperature.

As is shown in heats 1098, 1099, 1110, and 1116, in which the FeTi was in the furnace some time, the recovery of Ti is low unless it is added at the end of the heat. When this is done, the recovery seems to depend mainly on the temperature, very hot steel being required to take large amounts of FeTi into solution.

Segregation appears when the steel is too cold to dissolve the addition of FeTi rapidly, but is otherwise not troublesome. The recovery of Ti from the FeTi made by Thermit reduction is far higher than that of Zr from similarly made FeZr alloys. By analogy with Zr it seems possible that a SiTi alloy would be still more readily taken up by the steel. Petinot[35] has suggested an alloy containing Ti and Si in the rate of 5 to 1 as a deoxidizer for steel, because of the fusibility of $SiO_2$ and $TiO_2$ in the proportions given by the oxidation of this 5:1 TiSi alloy. This amount of Si would probably not be large enough to make any appreciable difference in the ease with which the alloy is dissolved by steel.

In regard to the solubility of ferrotitanium and its effect on steel, see Morehead,[36] Slocum,[37] Goldschmidt,[38] Fitzgerald,[39] Yensen,[40] and Hunter.[41]

## CERIUM STEELS.

Since cerium is in the same group in the periodic system as titanium and zirconium some experiments on adding cerium to steel were planned.

Before these were performed information was obtained through the courtesy of L. W. Spring, metallurgist of the Crane Co., Chicago, Ill., on some tests he had made, using Mix-metal obtained from the Fansteel Products Co., said to contain under 2 per cent of elements other than the cerium group, with about 50 per cent Ce, the balance being made up of the other elements of the group.

Moldenke[42] has studied the effect of adding 0.05, 0.10, and 0.15 per cent cerium metals to cast iron. He found that the transverse strength and the deflection were increased by the use of cerium and that the metal froze more slowly than untreated metal, thus giving less combined carbon; hence a softer, more readily machined metal is obtained.

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[35] Petinot, U., U. S. Patent 1260037, Mar. 19, 1918.

[36] Morehead, J. T., Discussion: Trans. Am. Electrochem. Soc., vol. 12, 1907, p. 79.

[37] Slocum, C. V., Titanium in iron and steel: Trans. Electrochem. Soc., vol. 20, 1911, p. 265.

[38] Goldschmidt, H., The melting point and its relation to alloying capacity: Met. and Chem. Eng., vol. 9, 1911, p. 348.

[39] Fitzgerald, F. A. J., Has titanium any influence on the properties of steel: Met. and Chem. Eng., vol. 13, 1915, p. 28.

[40] Yensen, R. D., Forgeability of iron-nickel alloys: Mining and Metallurgy, No. 157, sec. 9, Jan., 1920.

[41] Hunter, M. A., and Bacon, J. W., Some electrical properties of titanium alloys: Trans.

[42] Moldenke, R., Cerium in cast iron: Preprint of paper presented at 1919 meeting of the American Foundrymen's Assn.

Am. Electrochem. Soc., vol. 37, 1920, p. 391.

No figures are given to show whether there was any change in the percentage of sulphur or phosphorus, but he states that even when 0.50 cerium metals were added no cerium was found in the castings.

The Crane Co. tried Ce in red brass and in gray iron, and decided that it was injurious in brass (acting much like silicon) and too expensive to be of great promise in gray iron; then the company tried it in converter steel. Their results are shown in Table 7.

TABLE 7.—*Results of addition of cerium metals to converted steel.*

| Blow No. of heat. | Per cent Ce— |  | Analysis. |  |  |  |  | Physical properties.[a] |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  | Added. | Found. | Comb. C. | Si. | Mn. | S. | P. | Tensile strength. | Yield point. | Per cent elong. | Per cent red of area. | Fracture. |
| 1565 | None. | ........ | 0.37 | 0.42 | 0.84 | 0.086 | 0.054 | 107,000<br>90,250 | 60,600<br>51,900 | 21.5<br>22 | 18.4<br>31.2 | Gray cryst., fibrous. |
| 1565 | 0.5 | [b] 0.16 | .41 | .42 | .82 | .043 | .056 | [c] 68,500<br>98,050 | 47,500<br>51,250 | [c] 4<br>20 | 17.1<br>24.5 | Dirty brown and gray, fibrous. |
|  |  |  | Loss of S.......... |  |  | .043 |  |  |  |  |  |  |
| 1598 | None. | ........ | .40 | .32 | .75 | .088 | .056 | 90,400<br>89,000 | 50,500<br>45,500 | 20.5<br>20 | 23.4<br>25.8 | Two-thirds granular, fibrous. |
| 1598 | 1 | n. d. | .39 | .33 | .79 | .036 | .051 | [c] 83,250<br>91,100 | 49,500<br>46,550 | [c] 7<br>21 | ——<br>32 | Dirty, broke in shoulder, fibrous. |
|  |  |  | Loss of S.......... |  |  | .052 |  |  |  |  |  |  |
| 1643 | None. | ........ | .36 | .30 | .70 | .081 | .057 | 81,750 | 44,500 | 18 | 24.1 | Gray fir tree. |
| 1643 | 1 | .44 | .36 | .32 | .71 | .027 | .059 | 77,800 | 35,300 | 26 | 42.1 | Fibrous. |
|  |  |  | Loss of S.......... |  |  | .054 |  |  |  |  |  |  |
| 1826 | None. | ........ | .37 | .32 | .73 | .084 | .063 | 85,050 | 49,750 | 21 | 25.9 | Gray fir tree. |
| 1826 | 1 | n. d. | .36 | .33 | .72 | .041 | .063 | 97,250 | 53,600 | 25 | 32.2 | Gray and fibrous. |
|  |  |  | Loss of S.......... |  |  | .043 |  |  |  |  |  |  |

[a] Test bars made in keel-block form. All test bars annealed at 1,700° F. 7 hours and furnace cooled.

[b] Bureau of Mines analysis.

[c] First test bars on cerium-treated steels of heats 1565 and 1598 contained rust-colored inclusions, probably cerium oxide. Second bars cut from bottom of test block were free from inclusions.

The cerium was tossed into the stream of the metal while it was being poured from bull ladle to hand ladle. No violent reaction occurred when the cerium was added. The steel did not become "dead" as soon with cerium as without. A dross formed on top of the steel and came to the top of the riser of the mold.

As only 100 pounds of steel was used in each test, it had to be poured quickly to prevent chilling, not giving time enough for any dross formed to rise, as is shown by the dirty tensile-test bars on heats 1565 and 1598.

Microscopic examination of annealed samples showed that the steel to which cerium was added was far more uniform than the samples without it. The ferrite network usually formed by manganese sulphide inclusions was absent in the cerium-treated samples. These steels were not examined for inclusions in the unetched condition and the tiny inclusions, normal to a cerium steel, probably escaped notice in the etched sample.

Bars taken from the lower part of castings made from the cerium-treated steels were clean or at least free from macroscopic flaws; they showed marked superiority in ductility.

The most noteworthy feature was the consistent removal of S. Table 8 shows the results of four heats.

TABLE 8.—*Results of four heats of cerium-treated steel.*

| Heat No. | Ce added. | Per cent S before Ce added. | Per cent S after Ce added. | Per cent S removed. | Per cent S present removed. | Per cent Ce found. | Per cent recovery of Ce. |
|---|---|---|---|---|---|---|---|
| 1565 | 0.5 | 0.086 | 0.043 | 0.043 | 50 | 0.16 | 32 |
| 1598 | 1 | .088 | .036 | .052 | 59 | n. d. | .......... |
| 1643 | 1 | .081 | .027 | .054 | 67 | .44 | 44 |
| 1826 | 1 | .084 | .041 | .043 | 53 | n. d. | .......... |

Reducing the S from an average of 0.085 per cent to one of 0.037 per cent by a ladle addition is an interesting feat, and on account of the above data from the Crane Co., some attention was paid to the effect of Ce on S.

Some "Volcano" brand Mix-metal, said to contain approximately 5 per cent iron and 95 per cent cerium-group metals, was obtained in which the amounts of cerium-group metals was said to be approximately as follows:

| Cerium-group metals. | Per cent. |
|---|---|
| Cerium | 45 |
| Lanthanum | 25 |
| Neodymium and praseodymium | 15 |
| Samarium | 10 |

The lot received actually contained about 10 per cent Fe and 90 per cent of the cerium-group metals. In heats 1252 and 1253, the Mix-metal was thrown into the furnace after the melt had been skimmed, and stirred in. It floated and partly burned with sufficient violence to cause a small "popping" noise, some steel spatter being thrown about. Thereafter the Mix-metal was fastened to a bent iron rod by iron wire, and submerged in the steel, the operator standing at the side of the furnace, not in front of the door. The metal was stirred after the Mix-metal was added. In heat 1268, a silicocerium alloy, obtained through the kindness of Dr. Alcan Hirsch, was used. This was said to contain 26.7 per cent Fe, 30 per cent Si, 0.61 per cent C, 0.05 per cent S, 0.04 per cent P, and 42.3 per cent cerium metals.

Since the Armco iron used in making up the steels was already low in S (about 0.02 per cent) and very low in P (about 0.005 per cent), sulphur was added in the form of iron sulphide on heats 1256, 1257, and 1268. No aluminum was added to any of these heats, and the ingots were sound, as far as could be told on examining and drilling samples.

The results are given in Table 9.

TABLE 9.—*Results of treating steels with alloys of cerium group.*

| Heat number. | Total weight charged. | Weight sample poured. | Weight left in furnace. | Kw. h. used. | 1st or 2d heat. | Weight Ce alloy added. | Per cent Ce group metals— | | | | Per cent recovery Ce metals. | Per cent S— | | | | Remarks. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | Added.[a] | Found. | | | | Before Ce in sample. | After Ce in ingot. | | | |
| | | | | | | | | Top. | Butt. | Average. | | | Top. | Butt. | Average. | |
| | *Pounds.* | *Pounds.* | *Pounds.* | | | *Pounds.* | | | | | | | | | | |
| 1252 | 50 | ........ | ........ | 40 | 1st | 0.83 | 1.50 | 0.55 | 0.35 | 0.45 | 30 | ........ | ........ | ........ | ........ | A brownish orange slag, evidently containing cerium oxide, was formed, a little clinging to the furnace walls after the steel was poured, and more of it being continually formed in the ladle while the metal was held for degasification. This was noted in all the heats to which 0.5 to 1.5 per cent Ce was added. |
| 1253 | 50 | ........ | ........ | 37½ | 2d | .57 | 1.02 | .22 | .07 | .15 | 14 | ........ | ........ | ........ | ........ | Do. |
| 1256 | 56 | 5 | 51 | 37½ | 1st | .57 | [a] 1 | .06 | .06 | .06 | 6 | 0.155 | 0.068 | 0.066 | 0.067 | Metal was allowed to stand in ladle until almost too cold to pour well, to allow dross to rise. Strong odor of $SO_2$ was noted as soon as Ce was added to the metal, persisting until the steel was poured. Probably 2½ to 3 minutes elapsed between adding Ce and pouring it into the mold. |
| 1257 | 55 | 5.5 | 49.5 | 37½ | 1st | .27 | [a] .52 | .10 | .09 | .09 | 18 | .085 | .046 | .043 | .045 | 1½ minutes between adding Ce and pouring into mold. $SO_2$ odor was very faint. |
| 1258 | 50 | ........ | ........ | 32½ | 2d | .86 | 1.54 | 1.35 | .66 | 1 | 65 | ........ | ........ | ........ | ........ | |
| 1259 | 50 | ........ | ........ | 39 | 1st | .40 | .72 | .31 | .19 | .25 | 35 | ........ | ........ | ........ | ........ | |
| 1260 | 50 | ........ | ........ | 32½ | 2d | .01 | .02 | .012 | .008 | .01 | 50 | ........ | ........ | ........ | ........ | Ce was used as deoxidizer only, added in ladle. Metal was wilder than usual in ladle, and took longer to become dead, but the steel was sound. |
| 1268 | 55 | 2 | 53 | 40 | 1st | [b] .88 | .70 | .20 | .20 | .20 | 28 | .035 | .025 | .015 | .020 | Silicocerium was added in pea size and dissolved in steel readily. Not more than one minute passed between adding SiCe and pouring steel into mold. No $SO_2$ odor was noted. |
| 1272 | 50 | ........ | ........ | 32½ | 2d | .56 | 1.01 | .35 | .35 | .35 | 35 | ........ | ........ | ........ | ........ | |
| 1281 | 50 | ........ | ........ | 32½ | 2d | [c] .025 | .04 | ........ | .03 | ........ | 75 | ........ | ........ | ........ | ........ | Ce used as deoxidizer only in ladle, metal quiet at once after Ce was added. Steel was sound. |
| 1332 | 71 | ........ | ........ | 52½ | 1st | .115 | .91 | .54 | .22 | .38 | 42 | .030 | .024 | .026 | .025 | |
| 1333 | 71 | ........ | ........ | 52½ | 1st | .116 | 1.05 | .58 | .33 | .45 | 43 | .029 | .021 | .009 | .015 | |

[a] On basis of metal left in furnace.

[b] Silicocerium alloy.

[c] Part Mix metal, part CeFe alloy.

## RECOVERY OF CERIUM.

In all heats, except that in which steel 1375 was manufactured, where 0.10 per cent Ce or more was found in the finished steel, the fresh drillings had a noticeable odor, like acetylene, perhaps indicating that some, at least, of the cerium metals are probably present as carbide.

Appreciable segregation of Ce metals will be noted in several of these heats, and where this did occur more Ce was in the top of the ingot than in the butt, although the specific gravity of the cerium metals is not very much lower than that of steel. Addition of the Ce metals, in the form of ferrocerium or silicocerium, might possibly decrease segregation. None occurred in heat 1268, where silicocerium was used. When 0.7 to 1.5 per cent Ce was added to steel low in S, the recovery of Ce averaged about 33 per cent.

There was an elimination of S, as shown in Table 10.

TABLE 10.—*Elimination of sulphur from steels.*

| No. of heat. | Ce added. | S before Ce added. | S after Ce added. | S removed. | S present removed. | Recovery of Ce. | Time elapsed from addition of Ce to pouring into mold. |
|---|---|---|---|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* | *Minutes.* |
| 1256 | 1 | 0.155 | 0.067 | 0.088 | 57 | 5 | 2.75 |
| 1257 | .52 | .085 | .045 | .040 | 47 | 18 | 1.5 |
| 1268 | [a].70 | .035 | .020 | .015 | 43 | 28 | 1± |

[a] As silicocerium.

The more S there was removed the lower was the recovery of Ce. When a good deal of S was removed, there was a strong odor of $SO_2$, as if the S were taken out as cerium sulphide, which then rose as slag, some of the S being burned out in the air.

It was deemed possible that if the cerium were added to the molten metal and the metal held molten for a long time, the desulphurization might be more complete, therefore a couple of runs were made using as a base scrap steel containing about 0.10 to 0.11 per cent S and about 0.12 per cent P. More S was added as FeS. After the metal was melted, the furnace was opened, a sample poured, the Mix-metal added, the furnace at once closed tightly, and the heat continued. The data are given in Table 11.

TABLE 11.—*Tests showing effect of addition of cerium on desulphurization.*

| Heat No. | Total weight charged. | Total kw. h. used. | 1st or 2d heat. | Weight Mix-metal added. | Mix-metal added— | Time from adding Ce to pouring. | Weight sample poured. | Weight left in furnace. | Ce metals added.[a] | Ce metals found, per cent. | | | Recovery of Ce. | S before Ce added. | S after Ce added, per cent. | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | Top. | Butt. | Average. | | | Top. | Butt. | Average. |
| | *Pounds.* | | | *Pounds.* | | *Minutes.* | *Pounds.* | *Pounds.* | *Per cent.* | | | | *Per cent.* | *Per cent* | | | |
| 1287 | 55 | 40 | 1st...... | 0.62 | After 30 kw. h..... | 17 | 3 | 52 | 1.08 | None. | None. | 0 | 0 | 0.135 | 0.101 | 0.108 | 0.105 |
| 1288 | 55 | 42½ | 1st...... | .33 | .....do............ | 26 | 3 | 52 | .57 | None. | None. | 0 | 0 | .144 | .127 | .131 | .129 |

[a] On basis of metal left in furnace.

As soon as the Mix-metal was added to the steel in the furnace (in heats 1287 and 1288) a strong $SO_2$ odor was noted. While steel 1287 was being held in the ladle, a faint odor of $SO_2$ was noted, and while 1288 was held, the faintest odor was perceptible.

It was thought possible that in heats 1287 and 1288 the cerium might have been used up in eliminating phosphorus, since the scrap used was very high in P. Analysis showed, however that on both steels the sample taken before the cerium was added, the tops, and the butts of the ingot all contained the same amount of P—0.1 per cent.

In order to try cerium from more than one source, some "Cela iron" was obtained from the Fansteel Products Co. This was said to contain about 30 per cent iron, 70 per cent cerium metals (42 per cent cerium, 14 per cent lanthanum, and 14 per cent neo- and praseodymium). This was in shotted form, obtained by pouring the alloy into water. The Fansteel Co. stated that they believed this lot of alloy contained a good deal of oxide, due to shotting in water.

Two heats—1294 and 1295—were made as follows: Into the hot furnace was charged 51 pounds of scrap steel (such as was used in runs 1287 and 1288, but no extra iron sulphide was added), 4¾ pounds washed metal, and ¼ pound ferrosilicon.

After the metal was fully molten, a sample was poured for analysis, 3 pounds for heat 1294, 5½ pounds for heat 1295, and 0.83 pound Cela iron added and well stirred in. It went into the bath readily, without spattering. A few tiny flames or sparks were noted as it was stirred in. In heat 1294 the furnace door was at once put on, luted up tightly, and the arc started, every effort being made to keep a reducing atmosphere. In heat 1295 the door was not luted up, but the pouring spout and large cracks between door and furnace were kept open, and a slow stream of air was blown into the spout through an iron pipe, in order to keep the atmosphere at least partly oxidizing. In both tests nine minutes elapsed between adding the ferrocerium and pouring. Both runs were made exactly the same as to time and power consumption. A slight $SO_2$ odor was noted while heat 1294 was being poured and while the ladle was being skimmed. There was a thin slag on the metal. Both ingots were sound.

No final additions of any sort were made to heat 1294. The metal was perfectly quiet.

On steel 1295 there was noted only a trace of $SO_2$ while skimming from the ladle a little characteristic red cerium slag. On this heat the metal was wild, and 0.02 per cent Al was added to the top of the ladle after it had been poured. It was held as long as possible in the ladle and became practically quiet before pouring. It was poured before the ladle started to skull.

### COMPOSITION OF SOME STEELS WITH CERIUM ALLOYS.

Assuming that the scrap steel all had the composition shown by a random sample, which may not be true, as it was in rather large pieces not necessarily all from the same lot, the calculated composition of the steel is as shown below:

*Composition of steel 1294.*

| Constituents. | Calculated composition of sample. | 1294 found in sample. | Calculated composition of ingot. | Found in ingot.[a] |
|---|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* |
| C | 0.38 | 0.37 | 0.38 | 0.35 |
| Si | .19 | .21 | .19 | .18 |
| Mn | .40 | .41 | .40 | .41 |
| S | .10 | .104 | .10 | [b].033 |
| P | .11 | .102 | .11 | .105 |
| Ce metals | | | [c]1.07 | .04 |

[a] Samples of ingots 1294 to 1298 were made up of equal parts of drillings from top and butt.
[b] 0.067 removed.
[c] Added after taking sample; percentage of Ce metals in 1294 to 1298 is calculated on amount of steel left in the furnace after the sample is poured.

*Composition of steel 1295.*

| Constituents. | Calculated composition of sample. | 1295 found in sample. | Calculated composition of ingot. | Found in ingot. |
|---|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* |
| C | 0.38 | 0.27 | 0.38 | 0.25 |
| Si | .19 | .23 | .19 | .20 |
| Mn | .40 | .31 | .40 | .30 |
| S | .10 | .104 | .10 | [a].077 |
| P | .11 | .101 | .11 | .10 |
| Ce metals | | | [b]1.12 | None. |
| Al | | | [c].02 | n. d. |

[a] 0.027 removed.
[b] Added after taking sample.
[c] Added in ladle.

Two more heats were then made, using in each 50 pounds of Armco iron of less than 0.01 per cent P, of 0.037 per cent S, of only a trace of Si, about 0.01 per cent C, and about 0.02 per cent Mn. Washed metal sufficient to bring the calculated carbon to 0.37 per cent ferrosilicon, and ferromanganese to bring the Si to 0.2 per cent and the Mn to 0.41 per cent, were added when the heats were started. Iron sulphide was also added at the start in amount sufficient to bring the calculated S to 0.093 in heat 1296 and 0.063 in heat 1297. These were run on the same time and power schedule as heats 1294 and 1295, a sample being poured, the ferrocerium (Cela iron) added and stirred in, the door at once replaced and luted in, and heating continued nine minutes.

*Composition of steel 1296.*

| Constituents. | Calculated composition of sample. | 1296 found in sample. | Calculated composition of ingot. | Found in ingot. |
|---|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* |
| C | 0.37 | 0.22 | 0.37 | 0.26 |
| Si | .20 | .08 | .20 | 08 |
| Mn | .41 | [a].15 | .41 | [a].26 |
| S | .093 | [a].079 | .093 | .093 |
| P | .01 | n. d. | .01 | n. d. |
| Ce metals | | | [b].50 | None. |

[a] See note on steel 1298, p. 66.
[b] Added after sample was poured.

The metal was wild when the sample was poured. When the final steel was poured the metal was very hot and had a little red slag upon it and there was a very slight $SO_2$ odor when the furnace was opened, though none later. The metal was allowed to stand in the ladle and was quiet when poured. This was done before any skulling began. The metal was quiet in the mold until it began to freeze; then it effervesced violently and rose. The ingot was unsound clear to the butt. No Al or Ce was added to the ladle.

*Composition of steel 1297.*

| Constituents. | Calculated composition of sample. | 1294 found in sample. | Calculated composition of ingot. | Found in ingot. |
|---|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* |
| C | 0.37 | 0.16 | 0.37 | 0.24 |
| Si | .20 | .06 | .20 | .04 |
| Mn | .41 | .23 | .41 | .23 |
| S | .063 | .067 | .063 | .064 |
| P | .01 | n. d. | .01 | n. d. |
| Ce metals | | | [a].24<br>[b].03<br>.27 | .015 |

[a] In furnace. [b] In ladle.

This heat was run like heat 1296, save that it was rather wild in the ladle, and 0.03 per cent Ce metals (but no Al) was added to the top of the ladle and stirred in. The metal then quieted, but effervesced and rose in the mold just as steel 1294 did, and the ingot was unsound clear to the butt.

No odor of $SO_2$ was noted save a suspicion of an odor on knocking off the metal within the hot-top after the ingot had frozen.

Another heat was then made, using Armco iron and washed metal as before, but adding no iron sulphide, adding the same amount of ferrosilicon, but only about two-thirds the amount of ferromanganese, pouring a sample, adding 0.28 per cent Ce metals (as Cela iron), closing the furnace, running it nine minutes more, then adding the other one-third of the ferromanganese and an extra 0.10 per cent Si as ferrosilicon, besides 0.06 per cent Ce, to the metal in the furnace. Nothing was added to the ladle.

*Composition of steel 1298.*

| Constituents. | Calculated composition of sample. | 1298 found in sample. | Calculated composition of ingot. | Found in ingot. |
|---|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* |
| C | 0.37 | 1.01 | 0.37 | 0.25 |
| Si | .20 | .03 | .30 | .13 |
| Mn | .27 | [a].05 | .41 | .31 |
| S | .038 | .046 | .038 | .043 |
| P | .01 | n. d. | .01 | n. d. |
| Ce metals | | | [b].28<br>[c].06<br>.34 | .02 |

[a] This manganese figure is, of course, impossible, as are those for S and Mn on the sample taken in 1296, but all these figures have been checked. The samples were small, were a mass of blowholes, and show much oxidation. It hardly seems possible that the manganese was not diffused through the melt so that the sample poured from the top was not representative, nor that the discrepancy is due to oxidation of the sample, but these are the only explanations that have been suggested.

[b] 9 minutes before end.

[c] At end.

A faint $SO_2$ odor was thought to be detected only just after the addition of the 0.28 per cent Ce metals to the furnace. The metal was hot and quiet in the ladle and was poured just after slight skulling began.

The metal evolved some gas in the mold and showed signs of wanting to rise. It was just between effervescence and quietness. The ingot was full of blowholes at the top, but almost free from them at the butt.

In some previous runs (starting with Armco iron) on high-silicon, high-manganese nickel steels (1260 and 1281) in which about 0.20 per cent Si and 0.30 per cent Mn were added at the end to "swamp the gases," and 0.02 and 0.04 per cent Ce metals, respectively, were added in the ladle, to take the place of the usual 0.01 or 0.02 per cent Al as final deoxidizer, the steels contained, respectively, 0.01 and 0.03 per cent Ce metals and were sound, although steel 1260 gave off a trace of gas in the mold. Those runs made it appear that Ce was almost as good a deoxidizer as Al. Runs 1296 to 1298 indicate that it is not.

Considering heats 1294 to 1298, it is seen that in heat 1294, on the remelt of a steel previously containing its Si and Mn alloyed with it, the addition of 1 per cent Ce metals, allowed to act for nine minutes, reduced the S greatly. In heat 1295, like heat 1294, save that air was blown in after the Ce was added, which would tend to oxidize the Ce and use it up, a slighter, though marked desulphurization took place with 1 per cent Ce.

In heats 1296, 1297, and 1298, the iron had not been previously alloyed with Si and Mn, and there was a large loss of these elements. These three steels were quite evidently not sufficiently "killed" before the Ce was added, and no appreciable desulphurization took place, even though the last two contained small amounts of residual Ce.

### DESULPHURIZATION TESTS.

The steel must be carefully deoxidized and degasified, it appears, before the Ce is added. On this assumption, a material desulphurization without using some excess Ce seems unlikely, so that an appreciable amount would be left in the final steel. There would then be two questions remaining—first, how much Ce will it take to lower the S in a given steel from the given S content to the desired point, and how much Ce will remain in the steel? Second, how will this residual Ce affect the properties of the steel?

To test the desulphurization in a thoroughly deoxidized steel, another heat—1301—was made as follows: A steel calculating C, 0.37 per cent; Si, 0.20 per cent; Mn, 0.43 per cent; S, 0.066 per cent; P, 0.01 per cent; and Al, 0.03 per cent, was made up from Armco iron, washed metal, ferrosilicon, ferromanganese, and iron sulphide, melted on the usual schedule, and the furnace then opened, skimmed, and 0.13 per cent more silicon, 0.12 per cent more manganese, and 0.03 per cent aluminum added and stirred in, the sample then poured, 0.36 per cent Ce metals then added as Cela iron and stirred in, the furnace closed and run nine minutes more. The steel was then poured into the ladle onto an additional 0.02 per cent Al.

The idea was to "kill" the steel in the furnace by additions of ferromanganese, ferrosilicon, and aluminum before the cerium was added, leaving the cerium free to confine its attention to desulphurization and not to deoxidation. This heat was run on the same time and power schedule as the previous ones.

No $SO_2$ odor was noted at any time. Both the sample and the ingot were sound.

*Composition of steel 1301.*

| Constituents. | Calculated composition. | Sample. | Calculated. | Ingot. |
|---|---|---|---|---|
| | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* |
| C | 0.37 | 0.20 | | 0.31 |
| Si | .33 | n. d. | | n. d. |
| Mn | .55 | .40 | | .40 |
| S | .066 | .067 | | .064 |
| Ce | | | 0.36 | .01 |
| P below | .01 | | | |

In the preparation of the Ithaca-Halcomb series of Navy steels two cerium steels gave the following:

*Comparison of two cerium steels.*

| Heat number. | Calculated. | | | | | | Found. | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | C | Si. | Mn. | Ni. | S. | Ce. | C. | Si. | Mn. | Ni. | S. | Ce. |
| | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* | *P. ct.* |
| 1332 | 0.52 | 1.68 | 1.00 | 3.00 | 0.029 | [a] 1.50 | 0.43 | 1.77 | 0.78 | 2.78 | 0.024T<br>.026B | 0.54T<br>.22B |
| 1333 | .52 | 2.49 | .99 | 3.00 | .029 | [b] 1.64 | .43 | 2.54 | .91 | 2.86 | .021T<br>.009B | .58T<br>.33B |

[a] Half from Mix-metal, half from Cela iron.

[b] All Mix-metal.

These were cast into 6 by 3 inch molds, no hot-top being used. Steel 1332 weighed $70\frac{1}{2}$ pounds; steel 1333, 66 pounds. A faint $SO_2$ odor was noted after the Ce was added to steel 1333, but none was noted on steel 1332. The analysis shows a desulphurization on steel 1333, the sulphur being higher in the top than in the bottom of the ingot, whereas the desulphurization on steel 1332 is barely appreciable.

Allison and Rock [44] cite some tests on converter steel for castings to which 0.10 and 0.15 per cent cerium-lanthanum were added. The physical tests showed no favorable effect due to cerium. According to a private communication from Allison and Rock, there was no desulphurization.

In 1919, the Crucible Steel Co. made a few heats of cerium steels which showed myriads of tiny inclusions, supposed to be cerium-oxide spots. Samples were sent to Watertown Arsenal to have the count of the spots checked. The inclusions ran from 200,000 to 900,000 per square inch and were extremely tiny, globular, and disseminated all through the steel.

Through the Fansteel Co. data have been received on tests of Mix-metal in open-hearth steel made by a large steel foundry that does not wish its name used. The Mix-metal was said to contain 30 per cent iron-balance cerium.

These steels were of about 0.30 per cent C, 0.60 per cent Mn, 0.25 per cent Si, 0.04 per cent S, and 0.04 per cent P, with none, 1, 2, 3, 6, 8, and 12 ounces of cerium per ton; that is, 0.003 to 0.14 per cent cerium. The physical properties showed no effect due to cerium. It was stated that the cerium steels showed fewer inclusions, and larger ones than in the steel free from cerium, the cerium being thought to cause coalescing of the inclusions. The S content on the cerium-free steel was 0.043 per cent, whereas that on the cerium-treated steels, presumably from similar raw materials and similarly treated save for the final Ce addition, ran from 0.037 to 0.046 per cent. that is, no desulphurization is indicated.

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[44] Allison, F. G., and Rock, M. M., Studies of the microstructure of cast steel: Chem. and Met. Eng., vol. 23, 1920, p. 383.

No data on segregation of Ce have been secured from any outside source. Taking the average Ce as the mean of analyses from top and butt we have a recovery of 25 to 40 per cent on the average when the cerium is added last. When much over 0.3 per cent Ce is present there is, even in a small 50 to 100 pound ingot, a distinct segregation, cerium being higher in the top and lower in the bottom of the ingot. A similar segregation is the rule in zirconium steels.

No desulphurization has been noted when less than 0.5 per cent Ce was added in the Bureau of Mines experiments, and none of the outside tests in which Ce has been used in tiny amounts as a scavenger have showed desulphurization. With 0.5 per cent and above, the Crane test and several of the bureau tests show a marked desulphurization, but other tests which were attempted to be run uniformly with those that did show it show none at all.

When desulphurization occurs it appears to be a true desulphurization, not an error in analysis. The presence of Zr in steel, like that of Ti, causes low results in determining S by evolution, but the evolution and the gravimetric methods agree, as is shown by Table 11.

TABLE 12.—*Determination of sulphur by evolution and by gravimetric method.*

| Heat No. | S sample. | S by evolution. | S by gravimetric method. |
|---|---|---|---|
| | | *Per cent.* | *Per cent.* |
| 1256 | S sample | 0.156 | 0.155 |
| 1256 T | | .069 | .068 |
| 1256 B | | .067 | .066 |
| 1257 | S sample | .086 | .080 |
| 1257 T | | .051 | .046 |
| 1257 B | | .045 | .043 |
| 1268 | S sample | .037 | .034 |
| 1268 T | | .028 | .019 |
| 1268 B | | .014 | .011 |
| 1375 T | | .021 | .022 |
| 1375 B | | .013 | .009 |

## COOPERATIVE TESTS WITH WELSBACH CO.

In the cooperative tests with the Welsbach Co. steels were made in heats of about 90 pounds, the cerium (New Process Metals Co.'s Mix-metal) being added just before the melt was poured from the furnace, and the steel allowed to stand in the ladle until it was thoroughly quiet.

In polishing the cropped ends of the ingots of cerium steel made in the cooperative work with the Welsbach Co. for macro-etching, to see if there was any ingotism, it was discovered that one or more hair cracks had appeared in some ingots. The bottoms of the ingots were then cropped, polished, and examined as well.

The composition of this series of Ce steels was as follows:

TABLE 13.—*Cerium steels made in cooperation with Welsbach Co.*

| Heat No.[a] | Series No. | C. | Si. | Mn. | S. | P. | Ni. | Cr. | Ce.[b] | Ce used. | Results as to cracks. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1338 A | 4 | 0.38 | 0.32 | 0.71 | n. d. | n. d. | nil. | nil. | n. d. | 1.7 | Mass of fine cracks in center of butt, ingots discarded. |
| 1339 A | 5 | .38 | .35 | .68 | 0.016 T<br>.007 B | 0.008 | nil. | nil. | 0.61 T<br>.41 B | 1.7 | Very tiny cracks on top, hardly discernable; inclusions large; several very small cracks on butt. Ingot rolled. |
| 1347 A | 13 | .41 | .27 | .64 | .019 T<br>.007 B | .012 | nil. | 0.98 | .34 T<br>.32 B | 1.4 | Showed cracks on top, but not on butt. Ingots rolled. |
| 1347 B | 14 | .41 | .27 | .64 | .021 T<br>.007 B | .01 | nil. | .99 | .50 T<br>.41 B | | |
| 1352 A and B[c] | 19 | n. d. | n. d. | n. d. | n. d. | n. d. | 1.5± | .75± | n. d. | 1.3 | Both ingots showed cracks both on top and butt. Ingots discarded. |
| 1357 B | 25 | .43 | .35 | .58 | .017 T<br>.017 B | .008 | 2.47 | .95 | .22 T<br>.25 B | 1 | No cracks found on polished faces. After deep etching one hair crack appeared on ingot top. Ingot rolled. |
| 1363 [d] | 31 | .42 | n. d. | n. d. | n. d. | n. d. | nil. | 1± | n. d. | 1 | 6 by 3 inch ingot. Several approximately parallel cracks were found across the short width of the ingot top. Inclusions large. Ingot discarded. |
| 1365 A | 20 | .41 | .33 | .63 | .031 T<br>.006 B | .007 | 1.17 | .77 | .25 T<br>.25 B | 1 (B) | Tiny hair cracks on top and butt opening up on deep etching. Ingot rolled. |
| 1370 [e] | 36 | .36 | .34 | .66 | .012 T<br>.012 B | .012 | 2.46 | .92 | .18 T<br>.19 B | 1 | 6 by 3 inch ingot. No cracks. Inclusions mostly small. |
| 1374 A [f] | 52 | .54 | .48 | .84 | .013 T<br>.011 B | .006 | 1.02 | .84 | .42 T<br>.36 B | .6 | No cracks. |
| 1375 A [g] | 53 | .31 | .55 | .07 | .020 T<br>.009 B | .003 | nil. | nil. | .15 T<br>.13 B | 1 | Do. |
| 1376 A [h] | 54 | .30 | .65 | .06 | .032 T<br>.032 B | .005 | nil. | nil. | .04 T<br>.05 B | 1.1 | Do. |

[a] A means the first 3 by 3 inch ingot poured, B the second.

[b] Acknowledgment is made to J. P. Bonardi, formerly of the Bureau of Mines, for some of the cerium analyses.

[c] A and B ingots both cracked, no analytical determinations. Ni and Cr figures are amounts added, probably contains about 0.4 per cent C, 0.3 per cent Si, and 0.65 per cent Mn.

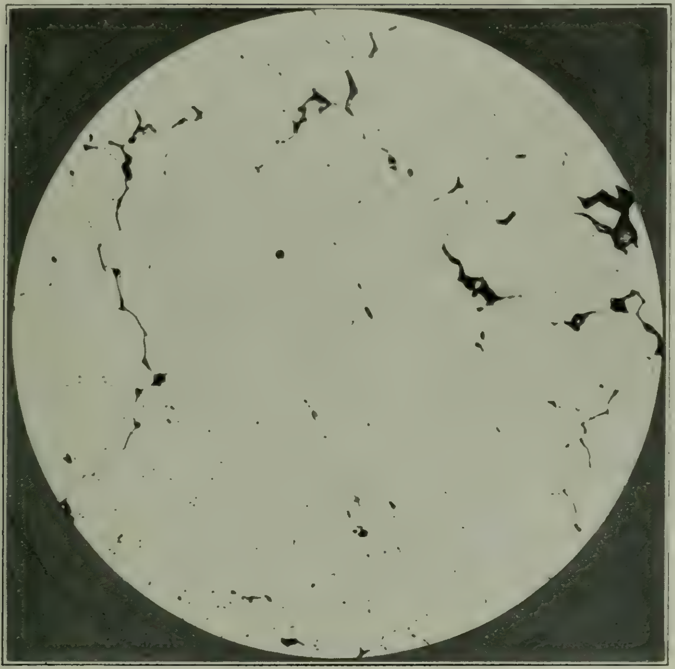
[d] Cracked ingot, C only determined. Cr figure is amount added, probably contains about 0.3 per cent Si, 0.65 per cent Mn, 6 by 3 inch ingot.

[e] 6 by 3 inch ingot.

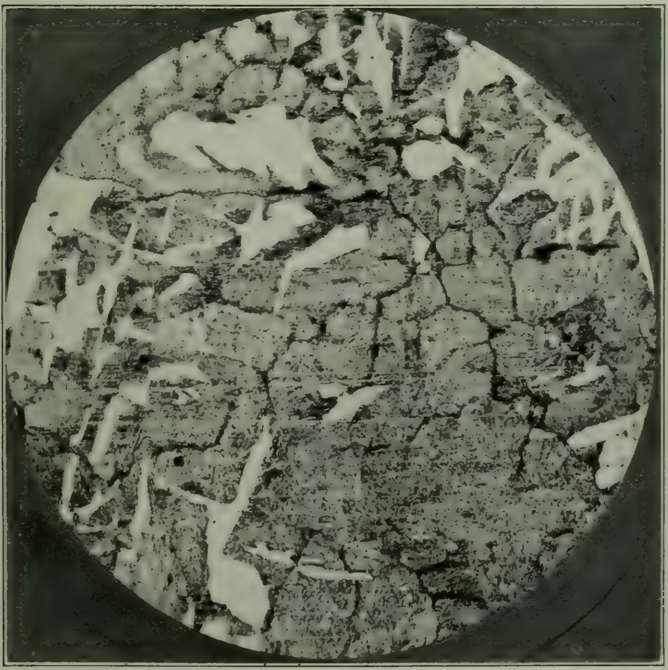
[f] This steel was poured cold, being dumped from furnace to ladle and from ladle to mold as rapidly as possible. It gave a high cerium recovery and did not crack.

[g] Calculated sulphur content of charge, 0.028 per cent.

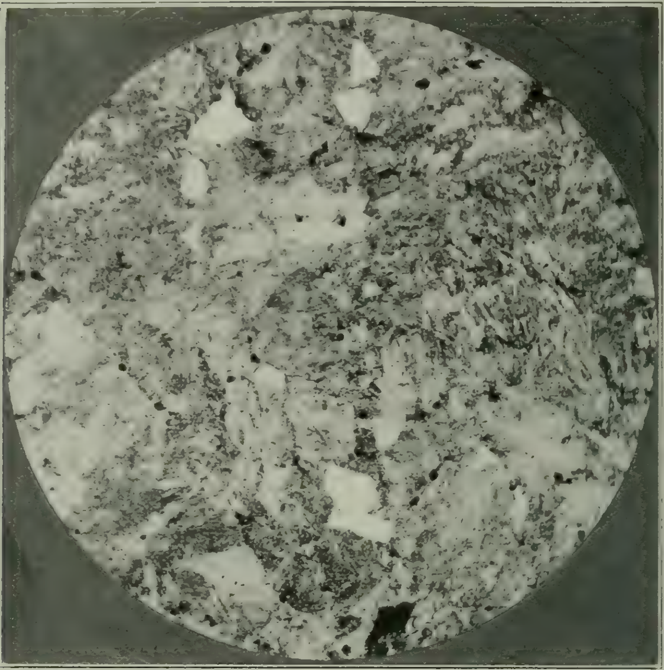
[h] Calculated sulphur content of charge, 0.069 per cent.



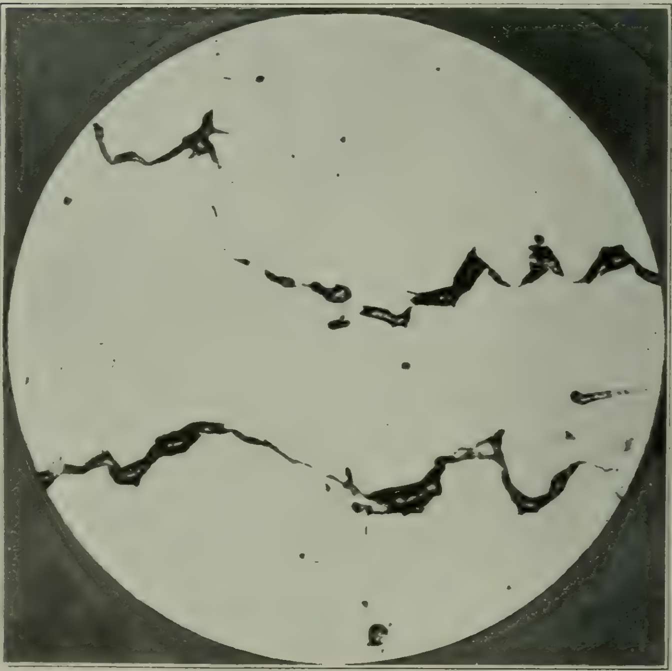
*A*. HEAT 1338, INGOT A, BUTT, CERIUM STEEL, UNETCHED, SHOWING CRACKS. MAGNIFICATION 100 DIAMETERS.



*B*. SAME AS *A*, $HNO_3$ ETCH, SHOWING CRACKS AT GRAIN BOUNDARIES. MAGNIFICATION 100 DIAMETERS.



*A*. HEAT 1347. INGOT A, BUTT, CHROMIUM-CERIUM STEEL. $HNO_3$ ETCH, SHOWING GLOBULAR INCLUSIONS MOSTLY AT GRAIN BOUNDARIES, BUT NO DEFINITE CRACKS. MAGNIFICATION 100 DIAMETERS.



*B*. HEAT 1347, INGOT B, TOP, CHROMIUM-CERIUM STEEL, UNETCHED, SHOWING CRACKS. MAGNIFICATION 100 DIAMETERS.

Heats 1338, 1339, 1347, and 1352, in which 1.7 to 1.3 per cent Mix-metal was added, were a trifle slushy. None of the cracks extended to the edges of the ingots, but appeared only at or near the center. None of the cracks were visible on a machined surface, and without magnification most of them were hard to detect on a ground and polished surface. The ingots with the tiniest cracks were rolled to ascertain whether the cracks would weld up in rolling.

On the ingots which did not crack there were globular inclusions ranging from very small ones, very close together, to much larger ones, farther apart. The tiny inclusions had coalesced in the cracked ingots to a greater or less extent, and nearly all globular inclusions were large. Most of the inclusions in the cracked ingots were, however, collected into the cracks, and appeared to be the cause of the cracks. These cracks appeared at the grain boundaries, and the material in the cracks is gray, looking like manganese sulphide. It would seem that there may be a cerium-manganese sulphide which has a low melting point, is the last thing to freeze, and is therefore mainly rejected to the grain boundaries, forming a line of weakness that causes a crack as the ingot solidifies and cools.

No cracks were found in any of the companion ingots which did not contain cerium, although quite a number were examined. These companion ingots contained around 0.65 per cent Mn and 0.017 to 0.038 per cent S, averaging about 0.025 per cent S; that is, they were much higher in S than most of the cracked ingots, particularly the butts of the ingots.

Desulphurization may be due to the formation of a readily fusible cerium sulphide (or cerium-manganese sulphide) which rises to the surface of the melt, burning in the air to $SO_2$ and cerium oxide. It may be due to some other cerium-group element in the Mix-metal. That there is a tendency for a sulphide to rise is clearly indicated by the fact that sulphur is often much lower in the butt than in the top.

The rising of this assumed complex sulphide may also account for the fact that the cracked ingots showed more and wider cracks on the top than on the butt.

Plates II, *A* and *B;* III, *A* and *B;* IV, *A* and *B;* and V, *A* and *B*, show typical cracks and inclusions.

Most of the cerium steels showed secondary pipe below the primary pipe, and had to be cropped deeper than other similar steels without cerium. This fact, combined with the trouble from cracking, the much dirtier appearance of the cerium steels than that of similar steels without cerium, the low recovery of cerium and its cost, all militate against the possibility of the commercial use of cerium, unless it confers exceptional physical properties. Nevertheless, the desulphurizing action of cerium or the cerium group of metals is interesting from the theoretical point of view.

### TESTS TO DETERMINE CONTROL OF SULPHUR BY CERIUM.

On the theory that cerium enters a complex cerium-manganese sulphide, it seemed possible that cerium might control sulphur just as manganese does, and do so in the absence of manganese. It was, therefore, thought worth while to make up a couple of steels, using cerium, and holding the manganese as low as possible.

Steels 1375 (series 53) and 1376 (series 54) were so made up, using Armco iron, washed metal, ferrosilicon, and Mix-metal.

*Calculated and found constituents of steels 1375 and 1376.*

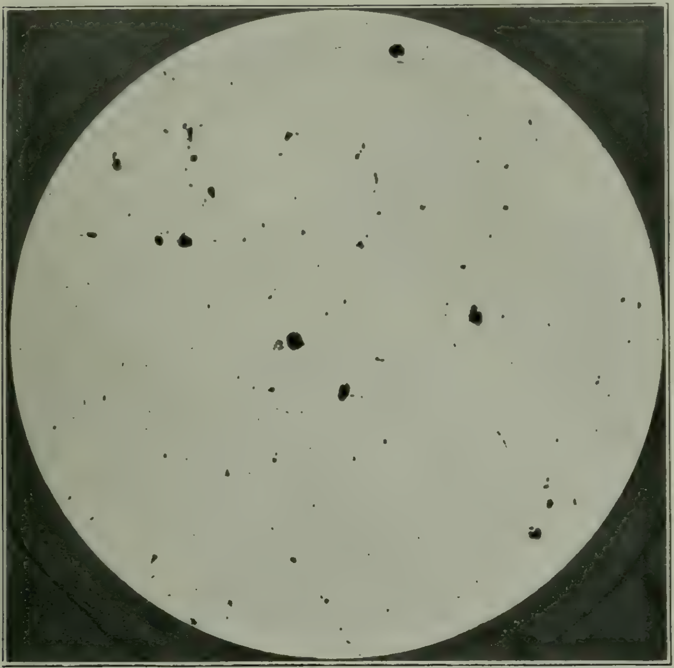
| Constituents. | Steel 1375. | | Steel 1376. | |
|---|---|---|---|---|
| | Calculated for no loss. | Found. | Calculated for no loss. | Found. |
| | *Per cent.* | *Per cent.* | *Per cent.* | *Per cent.* |
| C | 0.44 | 0.31 | 0.44 | 0.30 |
| Si | .40 | .55 | .40 | .65 |
| Mn | .03 | .05 T<br>.07 M<br>.08 B | .03 | .06 T<br>.07 M<br>.06 B |
| S | .028 | .020 T<br>.009 M<br>.009 B | 0.69 | .032<br>T, M, B |
| P | .007 | .003 | .007 | .005 |
| Cu | .03 | n. d. | .03 | n. d. |
| Ce | 1 | .15 T<br>.13 B | 1.10 | .05<br>T, M, B |

The furnace was thoroughly cleaned and fresh hearth lining put in before these steels were made. No aluminum was used. On account of the low Mn, the Si was raised a trifle to aid in deoxidation, and more Si was evidently reduced from the fire-clay bond in the fresh alundum-cement lining.

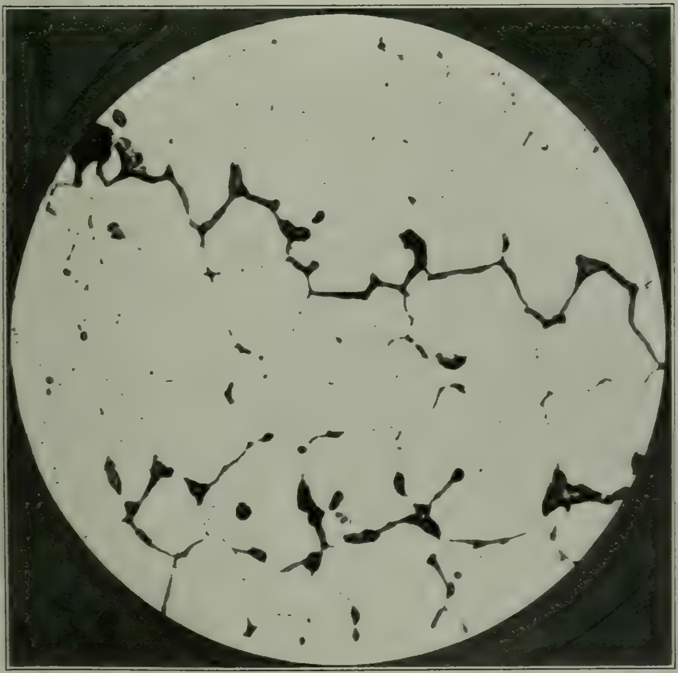
Steel 1375 had no S in addition to that contained in the raw materials, but iron sulphide was added to steel 1376 (with the rest of the charge). Half the ferrosilicon was added at the start. After the charge was melted and at pouring temperature, the melt was thoroughly skimmed, the other half of the ferrosilicon added, and the Mix-metal added and stirred in.

On steel 1375 one minute elapsed between adding the Mix-metal and pouring the mixture into the ladle and four minutes more before pouring into the mold. The metal was poured at 1,610°. No $SO_2$ odor was noted. The metal was thoroughly "dead."

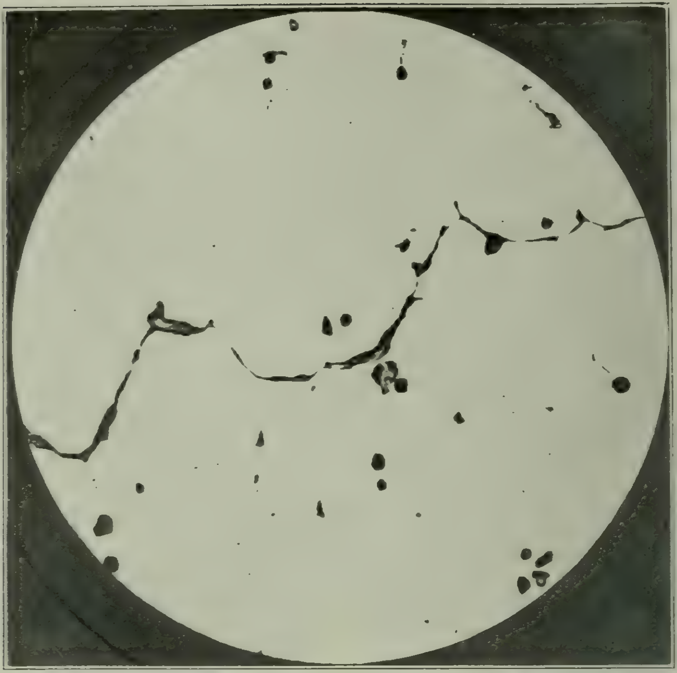
On steel 1376 one minute elapsed between adding the Mix-metal and pouring the mixture into the ladle and three minutes more before pouring into the mold. The metal was poured at 1,610°. A slight $SO_2$ odor



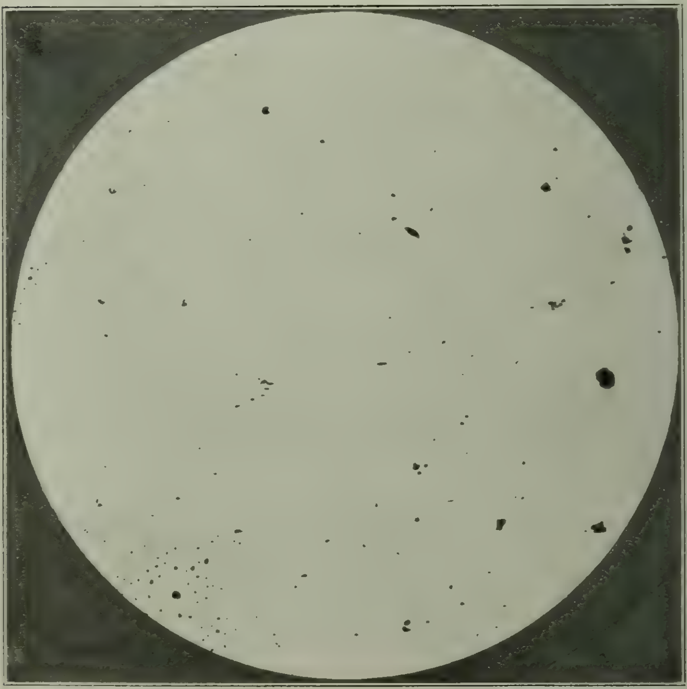
*A*. HEAT 1347, INGOT B, BUTT. CHROMIUM-CERIUM STEEL, UNETCHED, SHOWING INCLUSIONS AND ABSENCE OF CRACKS. MAGNIFICATION 100 DIAMETERS.



*B*. HEAT 1352, INGOT A, TOP. NICKEL-CHROMIUM-CERIUM STEEL, UNETCHED, SHOWING CRACKS. MAGNIFICATION 100 DIAMETERS.



*A*. HEAT 1363, TOP OF INGOT, CHROMIUM-CERIUM STEL, UNETCHED, SHOWING CRACKS. MAGNIFICATION 100 DIAMETERS.



*B*. HEAT 1370, TOP OF INGOT, NICKEL-CHROMIUM-CERIUM STEEL. UNETCHED, SHOWING INCLUSIONS AND ABSENCE OF CRACKS. NOTE PATCH OF TINY INCLUSIONS GROUPED ABOUT A LARGER INCLUSION LYING IN A SMALL AREA FREE FROM INCLUSIONS AS IF THE TINY INCLUSIONS WERE COALESCING WHEN THE STEEL FROZE. MAGNIFICATION 100 DIAMETERS.

was noted after the Mix-metal was added. The metal was thoroughly "dead."

Considering the analyses, there is found a normal or slightly greater than normal drop in carbon, a pick-up of silicon, probably from the lining, and a pick-up of manganese which is difficult to account for. The maker's analysis of the Armco iron was 0.015 per cent Mn. The Bureau of Mines analysis of a random sample gave 0.037 per cent Mn. The washed metal contained only a trace of Mn, on a random sample, as it was supposed to do. No Mn was found in the Mix-metal, and the ferrosilicon would have had to contain 5 per cent Mn to supply the picked-up manganese, which is out of the question. There was no steel left in the furnace from previous heats, as the inside lining was purposely chipped out and replaced by fresh.

The Mn analyses for the steel are correct, having been checked repeatedly for every sample (top, middle, and butt) by two methods. It seems probable that the Mn in the Armco must have run high.

This pick-up of Mn is regrettable, because it was hoped to secure a steel with Mn below the amount needed to combine with S as MnS.

The S found in steel 1375 requires 0.035 per cent Mn, top, and 0.012 per cent Mn, middle and butt, and 0.05 to 0.08 per cent Mn was present.

The S found in steel 1376 requires 0.055 per cent Mn, and 0.06 to 0.07 per cent was present, so that even in steel 1376 there was, at the end, a very small amount of Mn above that required to form MnS. Although more S was present in the charge than would form MnS with the Mn present, this Mn content is extremely low for a soft steel. In high-speed steel, manganese is held as low as possible, but even in such steel there is usually at least six times as much Mn as S. Levy, quoted by Sauveur,[45] says that on account of mass action, more Mn than corresponds to MnS is required to bind all the S as MnS and prevent the presence of FeS. Most metallurgists would expect steel 1376 to be decidedly red-short.

On steel 1375 the S came down from a calculated 0.028 to an average of 0.012, and on steel 1376 from 0.069 to 0.032.

The Ce recovery was very low, being only 5 per cent on steel 1376, in which more S was removed. These heats were held in the ladle quite a long time for so small heats (60 pounds), which probably tends toward a high loss of Ce.

The lower halves of each ingot were forged down from the 3 by 3 inch ingot to seven-eighths inch round under a steam hammer in a small local shop where they received no special care. They showed no sign of red-shortness and forged as well as any good 0.30 per cent C steel. The blacksmith said they acted like good stuff.

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[45] Sauveur, Albert, Metallurgy and heat treatment of iron and steel: Cambridge, Mass., 1918, p. 146.

There was no sign of cracks in the ingots, such as were noted in some of the previous Ce steels. These low Mn-Ce steels, 1375 and 1376, did not give out any odor when rubbed, as did the other cerium steels. With low manganese, then, the indications are that cerium not only eliminates some sulphur but controls the rest so that the steel is not hot-short. That is, one may apparently use Ce instead of Mn to control S. Such use is not very likely on account of expense, but the question arises whether a low Mn steel might not be useful for some purposes. Take steel 1376, for instance, with only about 0.06 per cent Mn and 0.05 per cent Ce. A regular 0.30 per cent C steel would have 0.35 per cent Mn and usually more.

According to Cushman and Gardner,[46] the presence of manganese may stimulate the corrosion of steel. Various other authorities have conflicting views as to the action of Mn, but it seems generally agreed that there is some interrelation among S, Mn, and Cu in steel, and that many factors in the corrosion of ordinary and copper-bearing steels are affected by this interrelation.

Although the presence of Ce introduces another variable, it would probably be possible to keep the residual Ce in the steel very low and to dispense with the Mn. Such a steel as 1376, which was "dead," sound, and not hot-short, with only a tiny amount of Mn and of Ce, offers a type of material by which the effect of Mn on corrosion might be studied. Cushman and Gardner say,[47] "Every metallurgist knows that steel which is free from manganese is difficult to roll, and that special care is necessary in order to shape it successfully." If this difficulty can be removed by the use of cerium, as it appears, such steel would be interesting material for the study of corrosion and the effect of manganese thereon.

## BORON STEELS.

To study the effect of boron in steel, some steels were made up to contain boron, using ferroboron of the following analysis, obtained from the Metal and Thermit Corporation: Lot 1—16.9 per cent B, 12.3 per cent Al, 8 per cent Si; lot 2—17.8 per cent B, 5.8 per cent Al, 0.9 per cent Si.

The data follow:

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[46] Cushman, A. S., and Gardner, H. A., Corrosion and preservation of iron and steel: New York, 1910, p. 78.

[47] Cushman, A. S., and Gardner, H. A., work cited, p. 126.

TABLE 14.—*Effect of boron in steel.*

| No. of heat. | Total weight charged. | Weight FeB used. | Lot 1 or lot 2 FeB. | Total kw. h. used. | 1st or 2d heat. | FeB added— | No. thermit cans used. | Size FeB. | Per cent B— |  |  |  |  | Notes. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  |  |  |  |  |  | Added. | Found. |  |  | Recovered. |  |
|  |  |  |  |  |  |  |  |  |  | Top. | Butt. | Average. |  |  |
|  | *Pounds.* | *Pounds.* |  |  |  |  |  |  |  |  |  |  |  |  |
| 1261 | 50 | 2.2 | 1 | 38 | 1st.... | At start....... | 0 | Hickory....... | 0.73 | 0.57 | 0.57 | 0.57 | 78 | Glossy borate slag formed, which fluxed lining somewhat. |
| 1262 | 50 | 1.35 | 2 | 40 | 1st.... | At end........ | 0 | Under 8-mesh. | .48 | .49 | .49 | .49 | 102 | Metal very hot, FeB dissolved rather readily but not so readily as judged by eye as SiZr. |
| 1263 | 50 | .68 | 2 | 40 | 1st.... | .....do......... | 0 | .....do......... | .24 | .28 | .32 | .30 | [a] 125 |  |
| 1264 | 50 | 1.35 | 2 | 30 | 2d..... | After 25 kw. h | 0 | Hickory....... | .48 | .46 | .54 | .50 | 104 |  |
| 1265 | 50 | .68 | 2 | 37½ | 1st.... | At end........ | 0 | Under 8-mesh. | .24 | .28 | .34 | .31 | [a] 129 |  |
| 1266 | 50 | 1.35 | 2 | 37½ | 1st.... | .....do......... | 0 | .....do......... | .48 | .44 | .43 | .44 | 92 |  |
| 1267 | 50 | 1.35 | 1 | 30 | 2d..... | .....do......... | 0 | .....do......... | .44 | .38 | .39 | .39 | 89 | Lot 2 FeB, with lower Al, appears to the eye to be dissolved more readily by the steel than lot 1. |
| 1274 | 50 | .31 | 2 | 37½ | 1st.... | .....do......... | 0 | .....do......... | .105 | .06 | .06 | .06 | 57 |  |
| 1275 | 50 | .31 | 1 | 36 | 1st.... | .....do......... | 0 | .....do......... | .105 | .08 | .08 | .08 | 76 |  |
| 1276 | 50 | .31 | 1 | 36 | 1st.... | .....do......... | 0 | .....do......... | .105 | .12 | .10 | .11 | 105 |  |
| 1277 | 50 | .31 | 1 | 35 | 1st.... | .....do......... | 0 | .....do......... | .105 | .10 | .09 | .10 | 95 |  |
| 1278 | 50 | .31 | 1 | 30 | 2d..... | .....do......... | 1 | .....do......... | .11 | ........ | .09 | .09 | 82 |  |

[a] The maker's analyses have been taken on the FeB. The alloy showed some signs of inhomogeneity. There is a slight segregation of B in heats 1263 and 1265. Both these causes may affect these recovery figures.

A very remarkable feature of the boron steels was that while the steel was freezing in the mold it had, for a very long period, a peculiar consistency. As the fluid started to cool it would first be like molasses, then like dough or modeling wax. Then the top of the ingot within the hot-top was poked to keep a hole open into the pipe for the escape of gas, the steel could be poked into any shape desired and the shape would be retained, acting as pie crust does when the fluted border is put on with the thumb.

This plasticity is retained far below the solidifying point of ordinary steel, apparently down to approximately the melting point of cast iron or even below. The steels poured fluidly, the plasticity shown in the mold apparently being due to a very different cause from the slushiness shown while pouring some of the steels high in uranium, which acted as if material was held in suspension.

Two other steels were made up, calculated to contain respectively 0.73 and 0.20 per cent B, and containing respectively 0.15 and 0.32 per cent C, which the Bureau of Standards attempted to roll. When the ingots were heated to the usual temperature for hot rolling, and were seized at one end by the tongs for lifting them from the preheating furnace, these two ingots dropped to pieces of their own weight. It was necessary to lower the preheating temperature materially in order to roll the other ingots of B steel. These two ingots were not analyzed for boron.

Even less than 0.1 per cent B seems to be sufficient, over a range of 0.15 to 0.7 per cent C, to produce the plasticity while freezing, and to render the ingots hot-short in rolling unless the rolling temperature is reduced from the normal.

It is probable that boron forms a very low melting eutectic with carbon, so that the range between the liquidus and solidus of a steel containing boron is enormously increased. In addition to work on the melting point of ferroboron, the Bureau of Standards has planned to determine the melting points, or melting ranges, of the boron steels made. The results of this work, when available, should throw light on the cause of the plasticity.

Boron appears to confer marked hardness on steel. If the other physical properties of the boron steels are good enough to be applicable to commercial use, their remarkable plasticity at temperatures below the melting points of ordinary steels might make it possible to utilize this characteristic.

## SUMMARY.

A cheap, experimental indirect-arc furnace has been designed and constructed for making up small experimental heats of alloy steels in which no refining slags are made. In this furnace some 375 heats of simple and complex alloy steels have been made in sufficiently close agreement with widely divergent chemical specifications.

The addition to steel of such alloying elements as tungsten, uranium, vanadium, chromium, molybdenum, nickel, silicon, manganese, zirconium, titanium, aluminium, cerium, boron, and copper has been quantitatively studied with especial reference to alloy recovery.

The recovery of tungsten, molybdenum, chromium, nickel, and copper, and of vanadium in amounts up to about 1 per cent, has been found to be practically quantitative when the ferro-alloy or metal is added to the furnace charge at the beginning of a heat.

With uranium, which is more easily oxidized than the elements mentioned above, the recovery has been shown to be somewhat erratic, but in general the best recovery was obtained by using a low-carbon ferro of 40 to 60 per cent uranium content, with the steel sufficiently superheated and the ferro added in the ladle or to the furnace after the heat is finished. Adding the ferro to the bath in the furnace before the heat was finished resulted in a very low recovery of uranium. The addition of much over 0.5 per cent was accompanied by marked segregation of uranium in the ingot.

With zirconium, the lowest recoveries were obtained when ferroalloys made by reduction of zirconium ore with carbon were used; often the recovery was practically zero. This does not check with experience reported by metallurgists of the Ford Motor Co. It should be noted, however, that there was a radical difference in the ferro-alloys used in the two groups of experiments, that used by the Ford Co. containing about 13 per cent zirconium, whereas none of the high-carbon ferros used in the experiments described above contained less than 44 per cent zirconium.

With ferrozirconium alloys, made by reduction of zirconium ore by the Thermit reaction alone, without electrical heating the recovery of zirconium was low, never being over 23 per cent and averaging about 10 per cent.

Nickel-zirconium alloys gave a better recovery of zirconium, an average recovery of 40 to 50 per cent being obtained when the alloy was added to the furnace at the end of the heat.

The best zirconium recoveries were obtained with alloys containing (*a*) about 30 per cent Zr and 45 per cent Si, and (*b*) 27 per cent Zr, 35 per cent Si, and 22 per cent Ni. These were very readily soluble in the steel bath when added at the end of the heat. Alloys of this type gave an average recovery of zirconium of 50 to 65 per cent. Steels containing over 0.3 per cent Zr generally showed marked segregation, more Zr being found in the top of the ingot than in the bottom.

Experiments with titanium showed that fair recoveries may be expected with a ferrotitanium made by the Thermit reaction, provided the ferro is added at the end of the heat. The steel should be very hot in order to secure rapid solution of the ferro.

The data show that the cerium metals, both in the form of cerium Mix-metal (silicocerium) and as ferrocerium, are very soluble in steel. The recovery probably depends largely on the length of the time the heat is allowed to stand after alloying, since a reddish-brown scum (assumed to contain cerium oxide) rose to the surface continually as the cerium-treated steels stood in the ladle. Segregation of cerium was noted in nearly all tests where more than 0.3 to 0.4 per cent Ce was present in the steel, more Ce being found in the top of the ingot than in the bottom.

The cerium alloys now available are not necessarily the most suitable for the introduction of cerium into steel.

The effect of the cerium group of metals on the sulphur content of the steel bath has been noted and a preliminary investigation made.

With Thermit-process ferroboron, containing about 17 per cent boron, uniformly good recoveries of boron were realized, averaging about 90 per cent. No marked segregation of boron was noted.

The peculiar properties manifested by boron steel during solidification have been noted.

Work on the molybdenum and on the cerium steels is still in progress and will be reported later.

## CONCLUSIONS.

The use of a small direct-arc electric furnace for making small experimental heats of alloy steel is attended with difficulty in the control of the carbon content.

Indirect-arc types, such as the Stassano, Rennerfelt, or the simple homemade furnace described herein, allow much better control of the carbon content. When a raw material is used sufficiently low in sulphur and phosphorus to obviate the making of refining slags, and straight melting, analogous to crucible-steel practice, is done, the making of special alloy steels in 50 to 100 pound heats, to chemical specification, is relatively simple in a 50-kilowatt indirect-arc furnace after the behavior of the ferro-alloys to be used is known, and the behavior of such alloys can be conveniently studied with the furnace.

This report deals only with the preparation of the various steels and with the recovery and the segregation of the different alloying elements. Some of the steels whose preparation is discussed in this section were of rather unusual composition, many being nickel steels high in silicon, or steels higher in various alloying elements than is common.

As the composition of many of the steels discussed herein is suitable only for special uses, no direct general conclusions can be drawn as to the value or lack of value of zirconium, titanium, uranium, boron, and cerium as true alloying elements in commonly used types of steels. The results so far obtained in various physical tests of the steels covered by this report, steels in which these elements are present in appreciable percentages, have not in general been promising; nor have they been as good as those of similar steels of the series without these elements. Recent reports of tests of plain carbon steels of low carbon content to which less than one-fourth of ·1 per cent of zirconium has been added are said to indicate that zirconium may have a beneficial effect, especially on notched-bar impact tests.

Even in very small ingots of 50 to 100 pounds, notable segregation of uranium, zirconium, or cerium generally appears when over about one-half per cent of the first, or about one-third per cent of either of the two latter is present in the steel.

These readily oxidizable elements are best added in the ladle, or to the furnace immediately before pouring. In remelting scrap containing these elements, they will be almost wholly lost, even in electric-furnace melting.

Uranium and especially zirconium alloys used to introduce these elements into steel should apparently be as free from carbon as possible. Alloys of zirconium with silicon or nickel, or both, give fair recoveries of zirconium, but no ferrozirconium tried does.

Titanium does not segregate as readily as zirconium, and its recovery from the ferro is higher; but in large amounts it is likely to segregate, and its recovery is not usually quantitative, although it may be quantitative if it is protected by the presence of a more readily oxidizable element, such as zirconium.

Although these elements are readily oxidized, none of them appear to be as efficient as aluminum or vanadium in degasifying "wild" steel. No special study has been made of very small amounts of these elements used as scavengers only, but all seem likely to leave their oxides or other compounds trapped in the steel as inclusions when added immediately before pouring, as is necessary in order to get a good recovery when using them as alloying elements rather than as scavengers.

Boron is almost quantitatively recovered when added at the end of the heat, and may be added at the start of the heat without great loss. In the amounts studied up to 0.6 per cent, no appreciable tendency to segregation was noted.

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